EFFECTS OF A SURFACE ENGINEERED METALLIC COATING ON ELASTOMERIC VALVE STEM SEAL LEAKAGE

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Valve stem seal leakage is a major source of fugitive emissions, and controlling these emissions can result in added expense in leak detection and repair programs. Elastomeric O-rings can be used as valve stem seals, and O-ring manufacturers recommend lubrication of elastomeric seals to prevent damage and to assure proper sealing.

In this research, a metallic coating was applied as a lubricant using a vacuum vapor deposition process to the surface of elastomeric valve stem seals. Valve stem leak measurements were taken to determine if the coated O-rings, alone or with the recommended lubrication, reduced valve stem seal leakage. This research determined that the metallic coating did not reduce valve stem leakage.

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CHAPTER 1

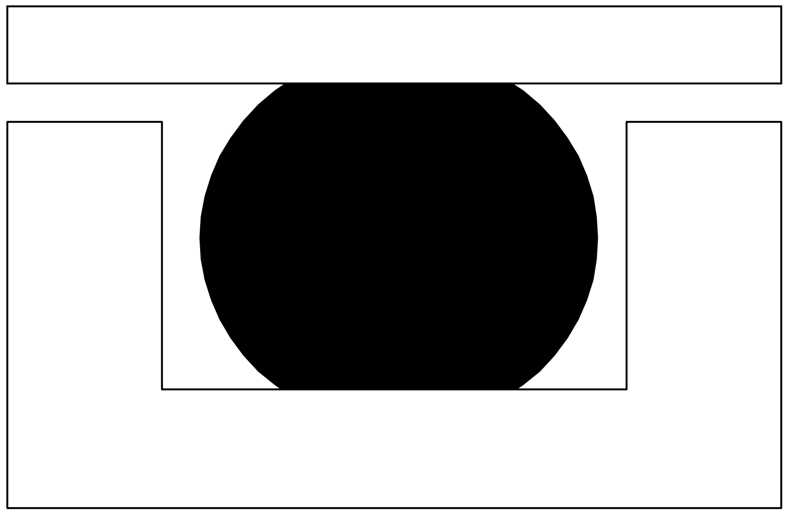
INTRODUCTION

Seals are used either to keep fluids contained or to prevent the intrusion of fluids. Seals can take the form of gaskets, O-rings, or packing. In the nuclear power industry, leakage from seals can result in environmental cost, safety and health cost, equipment failure cost, and maintenance cost (Electric Power Research Institute *Guide to optimized replacement of equipment seals* 1990). In particular, valve stem leaks require considerable maintenance in nuclear power plants (Electric Power Research Institute 1988).

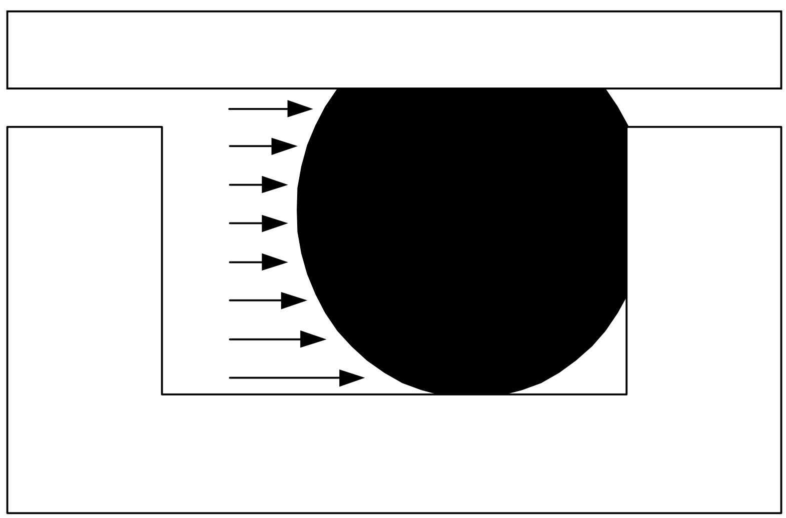
Chemical process industries (CPI) incur considerable expense in reducing volatile organic compound emissions. A significant portion of these emissions is from valve seals (Butcher 1997; Garrigues, Birembaut, and Ledauphin 1997; Harrison et al. 1995). Reducing volatile emissions through improved stem seals is important to power generating facilities as well (Ueda and Fujiwara 1997).

Elastomers are composed of very long chain organic molecules. These macromolecules generally have a random orientation. Because of their lack of order, they are technically classified as very viscous liquids with high surface tension.

Elastomeric seals function by flowing into mating surfaces (figure 1) and blocking the flow of a less viscous liquid (Parker Hannifin 1999). The surface of a seal must be able to adapt to the surface of the mating part. At the same time, the bulk of the seal must be stable under a compressive load (Römmler 1995).



O-Ring Seal Installed



O-Ring Seal Under Pressure

Figure 1. O-Ring sealing (Parker Hannifin 1999).

Elastomers, in general, have poor resistance to abrasion. Damage to an elastomer due to abrasion can reduce seal life (Parker Hannifin 1999). Poor surface finish of the mating part is a common failure mechanism in seals (Electric Power Research Institute *Guide to optimized replacement of equipment seals* 1990). Good lubrication, particularly during installation, can reduce abrasion and improve seal life. In dynamic seals, friction is also a major consideration. Seals and mating surfaces must be properly selected and maintained to reduce friction. Again, good lubrication is important. (Parker Hannifin 1999).

Surface engineering is the application of a thin film or coating to a substrate. A number of different industries use surface engineering as a common method of dealing with surface limitations (Bunshah 1994). One of the advantages of surface engineering is the ability to affect surface properties such as adhesion, friction, and galling without affecting bulk properties such as strength, hardness, and elasticity (Hopkins, Black, and Harrington 1997). TXU has developed surface engineering technology using a vacuum coating process. This process, now marketed as PlasmaBond®, has successfully applied a thin metal coating to metal surfaces. This thin coating acts as a dry film lubricant that reduces the potential for galling and reduces friction. Although the development of the TXU process has been focused on metallic parts, this process can be used to coat non-metallic items such as plastics and elastomers (Hopkins, Black, and Harrington 1997).

Problem Statement

The problem addressed in this research is valve stem leakage through an elastomeric O-ring seal. The valve used in this research is a carbon steel, pressure class

300, manual ball valve. This type of valve is the one of the most commonly used in TXU's natural gas transmission systems (English 1999). The TXU PlasmaBond® coating process was used to coat O-rings used as the upper valve stem seal. The metallic coating was applied using personnel, processes, and equipment at TXU's PlasmaBond® facility.

A method of reducing valve stem leakage that is easily retrofitted into existing equipment is an attractive option (Harrison et al. 1995). This research provided data necessary to determine if use of a metal-coated elastomeric O-ring seal in this type of valve should be pursued to reduce valve stem leakage.

Purpose

The purpose of this research was to determine if the application of a metal-coated O-ring would reduce leakage through an upper O-ring seal of a 2-inch, pressure class 300, manual ball valve. This research also determined if lubrication in conjunction with the metallic coating would reduce stem leakage.

Research Questions

There were three research questions addressed by this thesis. The first research question deals with the plasmabond treatment:

1. Will the use of elastomeric O-rings with PlasmaBond® metallic coatings reduce stem leakage?

This question has a corresponding null hypothesis 1 $(H_o)_1$: there is no change in leakage due to PlasmaBond® treatment. This is represented by equation 1 below where μ is the population average leak rate through the seal.

$$\mu_{\text{PlasmaBond}} = \mu_{\text{untreated}} \tag{1}$$

This research question also has an associated alternate hypothesis 1 $(H_a)_1$: there is less leakage due to the Plasmabond treatment. This is represented by equation 2 below.

$$\mu_{\text{PlasmaBond®}} < \mu_{\text{untreated}} \tag{2}$$

The second research question deals with the use of lubricant on the seal. This research question along with research question 3 helped differentiate the use of the PlasmaBond® as a lubricant from the effects of standard lubrication. Research question 2 was:

2. Will the use of a lubricant on elastomeric O-rings reduce stem leakage?

The corresponded null hypothesis 2 $(H_o)_2$ can be stated as follows: there is no change due to the presence of grease. This is represented as equation (3) below:

$$\mu_{\text{greased}} = \mu_{\text{ungreased}} \tag{3}$$

Likewise, alternate hypothesis 2 $(H_a)_2$ can be stated as follows: there is less leakage due to the presence of grease. This is represented as equation (4) below:

$$\mu_{\text{greased}} < \mu_{\text{ungreased}} \tag{4}$$

The final research question deals with the interaction between the two factors of plasmabond treatment and standard lubrication. This helped determine if using the PlasmaBond® along with the standard lubrication provided better sealing than the PlasmaBond® alone. The third research question was:

3. How does the PlasmaBond® treatment along with the use of a lubricant affect sealing capability?

This research question also has a corresponding null hypothesis 3 $(H_o)_3$: there is no interaction between the two factors. This is represented by equation (5) below where μ is the population average difference in leakage between the PlasmaBond® treatment and standard lubrication:

$$\mu_{AB} = 0 \tag{5}$$

Alternate hypothesis 3 $(H_a)_3$ can be stated as follows: there is an interaction between the two factors. This is represented by equation (6) below:

$$|\mu_{AB}| > 0 \tag{6}$$

Assumptions

The following assumptions applied to the research performed in this thesis:

1. Measuring and test equipment used in this research were assumed to provide accurate measurements because both leak testers were received from their respective manufacturer's shortly before the testing was conducted and all other measuring equipment was calibrated by TXU's calibration laboratory.
2. Seals used in this research were assumed to be homogeneous and a representative sample of seals used in this type of valve because the supplier of the seals is the authorized supplier of these valve parts and has a proven history of supplying products to TXU (English 1999).
3. Test valve clearances were assumed to be within manufacturing tolerances because the test valve is new, and the manufacturer of this valve has provided high quality valves to TXU (English 1999).
4. Installation techniques were assumed to be consistent with techniques used in the field because the recommend manufacturer's installation practice was followed for this research.

Limitations

This research had the following limitations:

1. Test was performed on only one type of seal material.
2. Only one test valve was used due to cost and test facility limitations.

3. Testing was conducted at ambient temperature.
4. No aging effects were included in the testing.
5. A non-flammable test gas was used due to safety considerations.
6. The test gas used may not directly correlate to natural gas under all conditions of pressure, temperature, and O-ring squeeze.
7. Testing was performed at TXU's Procurement Overview testing facility.

Overview of the Remainder of the Research

Chapter 2 provides a review of literature related to the thesis. Specifically discussed are valve seal related topics including valve stem seal types, valve stem leakage requirements, and valve stem leakage research. Chapter 2 also provides a discussion of elastomeric O-rings including the role lubrication plays in the function of an O-ring. Chapter 2 concludes with a review of literature for vacuum deposition technology, development of this technology at TXU, and related research.

Chapter 3 provides the details of the research including research design, control of variables, sample selection and treatment, test equipment, and testing methodology. The statistical basis for this research including sample size and objective criteria is provided as well.

Chapter 4 contains the testing results and analysis as well as observations made during the testing. The conclusions reached from this research are discussed in chapter 6, and chapter 7 contains recommendations for further research in this area.

CHAPTER 2

REVIEW OF LITERATURE

The review of literature focused primarily on: valve stem seal design and valve stem leakage requirements; elastomeric O-ring surface characteristics and seal leakage; and vapor deposition technology. Lastly, any relevant research related to this technology at TXU or that dealt with the reduction elastomeric seal leakage was reviewed.

Valve Stem Seals

The Electric Power Research Institute (EPRI) provides background information on valve stem seals (Electric Power Research Institute 1990). According to the EPRI guideline, there are two fundamental types of valve stem seals. Flexible seals rely on a flexible member, a diaphragm or bellows, to completely isolate the stem from the process fluid. The flexible member allows stem movement, but the stem does not slide through the seal. Flexible seals are very reliable but expensive. They are used when no leakage is permitted or when maintenance is not possible (Chopey 1998).

The other type of valve stem seal is packing. In this type of seal, the valve stem slides through the seal or packing. Radial pressure between the packing and the valve stem forms the seal. Packing is the more common valve stem seal because this type of seal can provide cost effective sealing even in severe service. In addition, this type of seal affords a wider range of stem movement than does a flexible seal.

Compression packing; lip-type, pressure-energized packing; and interference type seals are the basic categories of valve packing. These are shown in figure 2.

Compression packing is the simplest and most commonly used type of packing seal. This type of packing uses an external force, which is usually applied by a packing gland against the packing rings, to generate the radial force that forms the seal. The packing rings usually have a square cross section.

Lip-type packing uses a low external force and a pressure energizing action to generate the radial force that forms the seal. The pressure energizing action is a result of system pressure acting on the special shape of the packing. An increase in system pressure also increases the force at the sealing edge of the packing ring. Lip-type packing is also known as V-packing or chevron packing.

An interference seal uses elastic material and system pressure to provide the sealing force. O-rings are commonly used in this type of seal. O-ring seals used for valve stem sealing provide good sealing capability as well as a simpler and smaller seal design compared to other types of packing seals (Parker Hannifin 1999).

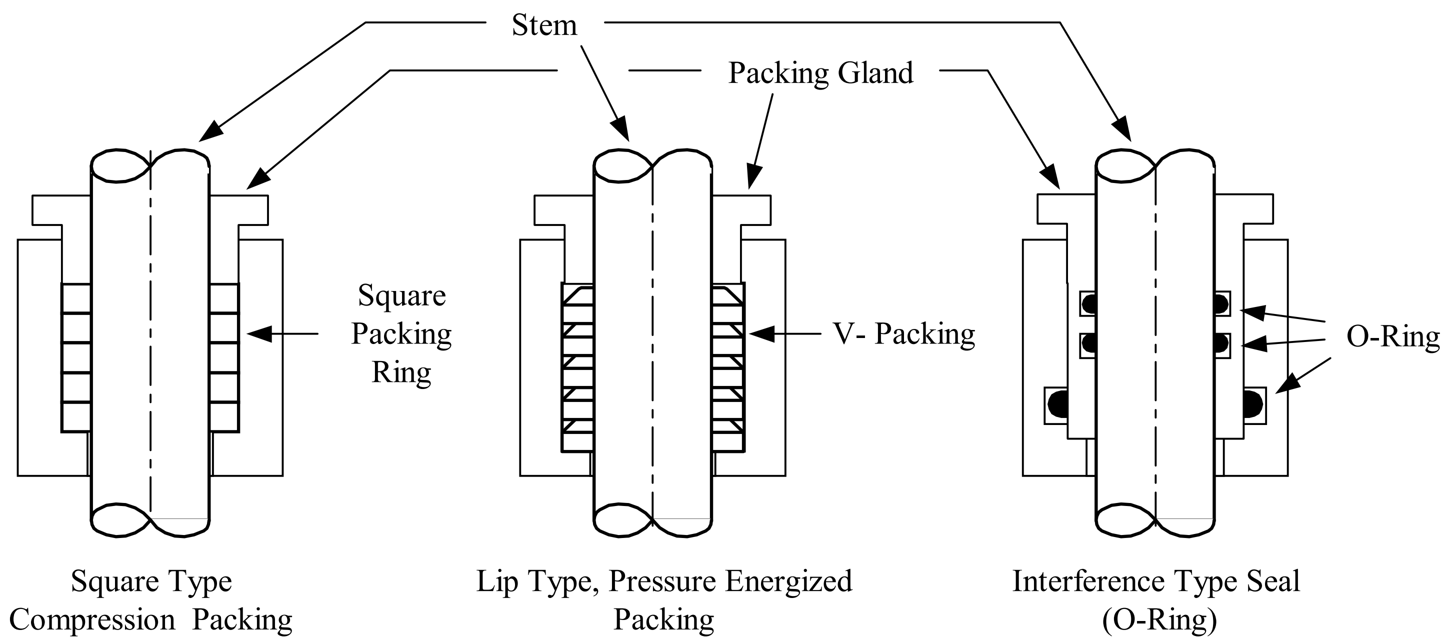


Figure 2. The three types of valve packing (EPRI 1990).

Valve Stem Leakage Regulations

Interest in valve stem leakage has been fueled in recent years by concerns over the effect that pollution has on the environment (Hoyes and Thorpe 1995). There are limits on the amount of chemicals that can be released to environment. In the United States, the Clean Air Act has restricted the amount of fugitive emissions that are permissible (Clean Air Act Amendments. U.S. Code 1990). Fugitive emissions are unanticipated or spurious leaks of chemicals from an industrial facility (Garrigues, Birembaut, and Ledauphin 1997). It is estimated that over half of the fugitive emissions of volatile chemical compounds are due to valve stem leaks (Butcher 1997).

Chemical process industry facilities are required under federal and many state regulations to have programs for fugitive emission leak detection and repair (LDAR). LDAR programs are aimed at reducing fugitive emission from pumps, valves, flanges, and other piping components. LDAR programs have four major elements: identify components subject to the program, conduct periodic monitoring of those components, repair any leaky components, submit reports to regulators (United States Environmental Protection Agency 1999).

Chemical process industries measure emissions at components as part of estimating the total amount of volatile organic compounds (VOC) or total organic compounds (TOC) released to the environment. The United States Environment Protection Agency (EPA) estimates that over 20 % of reported emissions from non-refineries and over 50 % from refineries are from fugitive emissions (United States Environmental Protection Agency 1999).

The definition of a volatile organic compound leak in federal regulations is 10,000 parts per million by volume (ppmv). Many states and local governments have defined leaks to a much lower level (United States Environmental Protection Agency 1999). Chemical process industries are required to monitor and report monthly if greater than 2% of their components are above the regulatory definition of a leak. Chemical process industries may monitor and report on a yearly basis if the number of components above the regulatory definition of a leak is less than 2% (U.S. Code of Federal Regulations Part 60: Standards of performance for new stationary sources 2000).

The EPA's National Enforcement Investigations Center (NEIC) conducted a study of seventeen refineries in 1999 to compare valve leaks reported by refineries with valve leaks measured by the EPA. While the reported proportion of leaking valves averaged 1.3 %, EPA testing found that on average 5.0 % of the valves at these facilities were at or above the regulatory definition of a leak. The EPA concluded that many of these facilities were releasing more chemicals to the environment than reported and this could result in an increase in local smog problems. It is worth noting that in this same study the EPA found that compliance to LDAR regulations is facilitated by using a lower than regulatory mandated definition of a leak. Several refineries used a leak definition as low as 500ppmv in their LDAR programs (United States Environmental Protection Agency 1999).

Valve Stem Leak Testing

Federal Regulations specify EPA Test Method 21 for VOC leak detection (U.S. Code of Federal Regulations Method 21: Determination of volatile organic compound leaks 2000). This method involves the use of a portable leak detector. The type of detector is not specified, however, the detector must be able to respond to the compound measured. Measurements using this test method are required to be in parts per million by volume. The instrument is also required to have a probe that sniffs the surface at potential leak locations. EPA Test Method 21 provides the following general directions for performing a survey of an individual source:

> Place the probe inlet at the surface of the component interface where leakage could occur. Move the probe along the interface periphery while observing the instrument readout. If an increased meter reading is observed, slowly sample the interface where leakage is indicated until the maximum meter reading is obtained. Leave the probe inlet at this maximum reading location for approximately two times the instrument response time. If the maximum observed meter reading is greater than the leak definition in the applicable regulation, record and report the results as specified in the regulation reporting requirements.
>
> **a.** Valves - Leaks usually occur at the seal between the stem and the housing. Place the probe at the interface where the stem exits the packing and sample the stem circumference and the flange periphery. Survey valves of multipart assemblies where a leak could occur.

Most of the recent research found that is related to valve stem seals is based, in part, on EPA Test Method 21. Laboratory testing usually involves testing a valve, or a valve stem and seal assembly at operating conditions. Instrumentation typically measures leakage in parts per million by volume (ppmv) or can be correlated to an equivalent reading in ppmv.

One of the major differences between laboratory testing and EPA test method is the test gas used. The regulatory definition of a leak for many compounds is referenced to instrumentation calibrated for methane. Due to safety considerations, most laboratory and manufacturer tests use an inert gas. This is typically helium (Garrigues, Birembaut, and Ledauphin 1997). It is generally accepted that ppmv leak measurements using helium need to be double to approximate the leakage using methane (Lowe 1995). Leefe and Davies performed a study of the correlation of valve stem leakage between methane, helium, and steam. Their study showed that while there was considerable scatter on a point by point basis, there was a reasonable correlation between methane and helium if the results are averaged over a number of tests (Leefe and Davies 1995).

Lowe conducted laboratory testing of standard compression asbestos and special graphite packing materials (Lowe 1995). He first tested the packing in a test assembly that consisted of a stem and seal assembly mounted below a control valve operator. Helium was used as the test medium. He found that leakage from the asbestos packing could not be reduced below 10,000 ppmv despite repeated tightening of the packing. The graphite packing had a much better performance. Initial leakage was less than 50 ppmv. After 100 cycles, leakage had reached 200 ppmv. The testing was concluded after 4000 cycles when the leakage had reached 10,000 ppmv. Lowe also tested a number of various size stock gate and globe valves. Several manufacturers provided these valves. Helium was again used as the test medium. Each of these valves was tested at 7, 20, and 40 bar. At each pressure plateau, each valve was manually operated full open to closed. Lowe found that most of these valves had leakage that was significant (well above 1000 ppmv). The highest measurement was 80,000 ppmv. Note that the leakage using methane would have been even higher. Each valve was then repacked with the special graphite packing. Leakage was dramatically reduced. Most of the re-packed valves had no detectable leakage. The highest detectable leakage was 150 ppmv. Lowe concluded that re-packing could reduce stem leakage despite existing clearance variations and stem damage.

BP Research and Engineering tested a number of different graphite packing products and standard asbestos compression packing (Harrison et al. 1995). For this research, they used two control valves that had been removed from process service in their testing. In addition, several different valve stems of varying degrees of wear were used in the testing. This was to simulate the range of plant conditions that may be encountered in retrofitting packing. The tests were conducted using methane as a test gas and leak measurements were performed in accordance with EPA Test Method 21. The control valves were initially cycled 100 times at a pressure of 7 bar. Pressure was then increased to 20 bar and the valves were cycled an additional 100 times. Pressure was then raised to the final test pressure of 40 bar and the valves were then cycled at a rate of 6 cycles a minute. The valves were cycled at this rate for a period of an hour followed by a rest period of 15 minutes. The cycle and rest periods were repeated until conclusion of the test. Testing was concluded when valve stem leakage exceeded 10,000 ppmv after one re-torquing of the packing gland. BP found that graphite packing had much lower leakage than the standard asbestos packing. The initial leakage for the asbestos packing was well over 1000 ppmv, and the test concluded at 200 cycles. The graphite packing initial leakage ranged from approximately 1 to 100 ppmv. The graphite packing tests were concluded after 3000 to 9000 cycles. From this testing, BP Engineering and Research determined that valves at BP Chemicals' facilities should be repacked using the improved graphite packing because re-packing would significantly reduce emissions.

They projected cost savings from reduced emissions in maintenance and product losses. Subsequent to the testing, BP Chemicals implemented a re-packing program.

Ueda and Fujiwara compared improved and conventional graphite packing products that are used for valve stem seals in volatile fluid service (Ueda and Fujiwara 1997). Their testing involved a stem and packing seal assembly that simulated a reciprocating valve stroke. Packing was pressurized to 5 MPa using helium. Stem leakage, packing gland tightening stresses, and frictional forces were measured for each of the different packing products. For each packing material, the stem was cycled 500 times. Leakage was measured from a seal housing leak-off connection. Improved packing had virtually no leakage through all of the cycles. Conventional packing initially had no leakage, however, at 100 cycles leakage occurred at a rate of 0.25 ml/min. This leakage increased to 0.55 ml/min at 500 cycles. In this test setup, leakage of 0.3 ml/min corresponds to roughly 500 ppmv methane. Ueda and Fujiwara concluded that the important properties of valve stem packing material used in volatile fluid service are the ability to block minute permeation and leakage of gases, and the abrasion resistance of the packing material under dry sliding conditions.

Published valve stem leakage literature discussed so far has dealt with linear valves. These are valves in which the valve stem travels in a linear fashion in a direction along its major axis. Globe and gate valves are examples of linear valves. The other category of valve used for control and isolation is the quarter turn valve. In a quarter turn valve, the valve stem rotates about its major axis one quarter of a revolution to go from full open to full shut or full shut to full open. Ball, butterfly, and plug valves are examples of a quarter turn valve (Electric Power Research Institute 1990). Quarter turn valves have become more popular recently, in part, because they inherently have fewer emissions (Chopey 1998). It has been estimate that 5% to 15% of the gate and globe valves with asbestos packing could be expected to leak while only 2% to 3% of ball valves would be expected to leak (Butcher 1997).

In the review of literature for valve stem fugitive emissions, only one study that evaluated stem leakage for a rotary valve seal was found. Leeds University tested a new lip-type graphite packing material developed by the Flexitallic Corporation (Hoyes and Thorpe 1997). This testing used helium pressurized to 40 bar as the test gas, and leakage measurements were performed using the methodology of EPA Test Method 21. A 2-inch, class 300, valve body; a valve stem; and a stem seal assembly were used to perform this testing. The valve stem was configured to allow attachment of either a linear or a rotary actuator. This new packing was tested for 10,000 cycles as either linear or rotary packing. The testing showed no appreciable difference in the leakage for the linear packing (280 ppmv) compare to the rotary packing (200 ppmv).

Elastomeric O-Rings

Elastomeric O-rings have been used for sometime in seals for a variety of products. As discussed earlier, elastomeric O-rings are commonly used in interference type packing seals because the use of O-rings results in a good seal that is simpler and smaller.

Any seal functions by blocking passages that could allow a fluid to escape. An elastomeric O-ring, a very viscous liquid with high surface tension, is forced by mechanical (squeeze) and fluid pressure to flow into clearance between the stem and gland, and thus block the potential leakage path. The elastomer should not actually flow into the clearance but rather block it (chapter 1, figure 1). When the O-ring seal is at its pressure limit, the pressure causes the O-ring to begin to flow into the clearance and the O-ring is said to be extruding. Any further increase in pressure causes the O-ring to flow through the clearance. The surface of the O-ring no longer blocks the process fluid, which results in O-ring extrusion failure (Parker Hannifin 1999).

O-ring seals can be broadly classified as either static seals, which have no relative motion between the members to be sealed or dynamic seals which have relative motion between the members to be sealed (Martini 1984). Dynamic seals are a more demanding application of an O-ring because of the friction and wear considerations between the O-ring and components to be sealed. Valve stem seals are dynamic seals (Parker Hannifin 1999).

O-Ring Lubrication

Lubrication of an O-ring is one the most important considerations. Lubrication is particularly beneficial during the installation process. Lubrication helps prevent abrasion, cutting, and pinching that can damage the O-ring during installation. Lubrication also aids in properly seating an O-ring during installation.

Lubrication is also very important for the operation of a dynamic seal. Elastomers have a high coefficient of friction with metals. Friction has two components, running friction and breakout friction. Running friction occurs during relative movement of the seal. Lubrication helps physically separate the asperities in the surfaces of the metal and the elastomer. The lubrication film also helps control chemical reactions at the boundary layer between the elastomer and metal. These chemical reactions can result in the formation of particles that damage the surfaces and limit seal ability (Martini 1984). Breakout friction is the other type of friction that occurs in O-ring seals. Breakout friction occurs after the seal has been at rest and first starts to move. This friction is a function of how long the seal has been at rest and is usually much higher that running friction. It is believed that this is due to the elastomer flowing over time into the voids and crevices in the surface of the metal (Parker Hannifin 1999). Also, lubrication tends to be squeezed out during periods of rest so there is very little lubrication initially (Hörl and Haas 1997). Lubrication has not been as effective in reducing this friction (Parker Hannifin 1999).

O-ring lubrication is also important in pneumatic seals. When a seal is used for liquid service, the process medium provides some lubrication. In the case of gas service there is less inherent lubrication from the process fluid so the initial lubrication of the O-ring becomes more critical. Besides the reduction in friction, lubrication can reduce the leakage of a gas through the O-Ring.

Elastomers are permeable and allow to some extent air, gases, and volatile liquids to diffuse through them. The rate of diffusion through the elastomer is based on the type of elastomeric material and the specific gas or vapor involved. There are, however, other factors that also affect the leak rate. Parker (Parker Hannifin 1999) provides a rough approximation in equation (7) below:

$$L = 0.7 * F * D * P * Q * (1-S)^2 \tag{7}$$

Where:

L is the leak rate in cc/sec.

F is the gas permeability through an elastomer at a given pressure.

D is the inside diameter of the O-ring in inches.

P is the differential pressure across the O-ring in psi.

Q is an empirical factor that is dependent on squeeze and lubrication.

S is the squeeze of the O-ring expressed as a decimal.

Note that this formula is only a rough approximation of leakage that occurs as a result of a gas permeating through the O-ring and not leakage at the sealing surface. Lubrication has a direct influence on this leakage. Parker provides a graph that shows this relationship which is condensed into table 1.

Table 1. Q factor versus squeeze for lubricated and unlubricated O-rings (Parker Hannifin 1999)

	Factor Q	
Percent Squeeze	Dry Ring	Lubricate Ring
15	1.5	0.72
20	1.35	0.72
25	1.2	0.72
30	1.15	0.72
35	1.10	0.75
40	1.06	0.8
45	1.03	0.9
50	1	1

As can be seen from the table, lubrication has a big influence on the leakage through the O-Ring, particularly at lower squeeze levels. Dynamic seals have lower squeeze because of friction and wear considerations. Typically dynamic seals are squeezed in a range of 8 to 16%, and for small O-rings it may be permissible to go up to a squeeze of 25% (Parker Hannifin 1999).

Surface Engineering

The semiconductor industry has for some time used surface engineering technology to apply thin metallic layers to the surface of semiconductors (Baliga 1997). Plasma based coating processes have been used to apply wear resistant coatings to machine tools and are being developed for more widespread use on other metallic parts (Mathews et al. 1995). Plasma based coatings are also being applied as potential protective coatings to plastics exposed to fast atomic oxygen such as encountered in low earth orbit (Kleiman, Iskanderova, and Tennyson 1998). Plasma based coatings are being developed for application on plastic packaging material as a potential solution to the permeation of oxygen and water vapor, a common problem in the food, beverage, and pharmaceutical industries (da Silva Sobrinho et al. 1998).

One of the advantages of surface engineering is the ability to affect surface properties such as adhesion, friction, and galling without affecting bulk properties such as strength, hardness, and elasticity (Hopkins, Black, and Harrington 1997). Although there has been extensive development in the application of coatings to metals, semiconductors, and plastics (Yang et al. 1997), there was little published literature found discussing the development of surface engineering in the area of elastomers.

Polymer Coating Limitation

Substrate materials composed of organic polymers, such as elastomeric seals, offer unique challenges to the application of a metallic coating using processes currently used for metallic and semiconductor substrates. Organic polymers have lower temperature limits during processing than do most metals or semiconductors. During many of the physical vapor deposition processes (discussed in the next section), the substrate reaches temperatures of approximately 500° C. In chemical vapor deposition, which is also used to deposit a thin layer on semiconductors and metals, the substrate can reach temperatures of nearly 1000° C (Dini 1997). These temperatures would be detrimental to polymers (Meier 1996).

Some current metallic coating processes rely on the conductivity of the substrate to establish a negative charge or bias directly on the substrate. Positively charged ions are attracted to the negatively charged substrate as part of the coating process (Dini 1997; White 1995). Most polymers are nonconductors, therefore, a negative charge cannot be established directly on the substrate (White 1995).

Some polymers contain volatile molecules such as water or oils to remain supple. These volatile molecules out gas when exposed to a vacuum (Harrington and Kidd 1998). Out gassing can interfere with a vacuum process such as vacuum deposition. Polymers that contain volatile molecules may not be suitable for coating using vacuum deposition (Society of Vacuum Coaters 1998).

Vacuum Deposition Technology

Deposition involves producing a solid from a vapor phase. The properties of a material produced from a vapor phase can be varied much more than material formed from a liquid phase. Deposition processes involving a vacuum can be categorized into two broad categories, chemical vapor deposition (CVD) and physical vapor deposition (PVD). In CVD, reactant gases are introduced into a vacuum chamber. A chemical reaction takes place at the substrate, and a product of this reaction is deposited on the substrate. CVD generally involves heating the substrate to fairly high temperatures. The other vacuum deposition category, PVD, is a physical process and does not involve a chemical reaction (Bunshah 1994).

Physical Vapor Deposition

PVD is a surface coating process in which the material is atomistically deposited. The depositant material is vaporized, transported through a vacuum or plasma, and condensed on a substrate. PVD is used to apply elemental and molecular coatings with thickness ranging from a few nanometers to several microns. While there are a number of variations and special processes, the three primary PVD methods are vacuum evaporation, sputter deposition, and ion plating (Bunshah 1994; Society of Vacuum Coaters 1998).

Vacuum Evaporation.

Vacuum Evaporation was the earliest and is the simplest form of PVD (White 1995). This process involves heating the depositant material to produce a vapor. The thermalized vapor travels through a vacuum until it reaches the substrate where it condenses. The vacuum environment assures there is no gas scattering of the vapor during its travel. The vapor travels in a line of sight trajectory from the vapor source to the substrate. The line of sight trajectory allows the use of masks to limit areas of deposition, however, the straight-line trajectory limits the ability to coat complex shapes (Society of Vacuum Coaters 1998). The energy of the vapor atoms is relatively low, 0.1 eV to 0.5 eV (Bunshah 1994). The low energy of the vapor atoms causes limitations on the ability of the coating to adhere to the substrate (White 1995). Vacuum evaporation can result in high deposition rates compared with other methods, and this method usually involves heating of the substrate (Bunshah 1994). Figure 3 shows a simple vacuum evaporation arrangement. Sputter deposition gained industry attention because of the limitations associated with vacuum evaporation (White 1995).

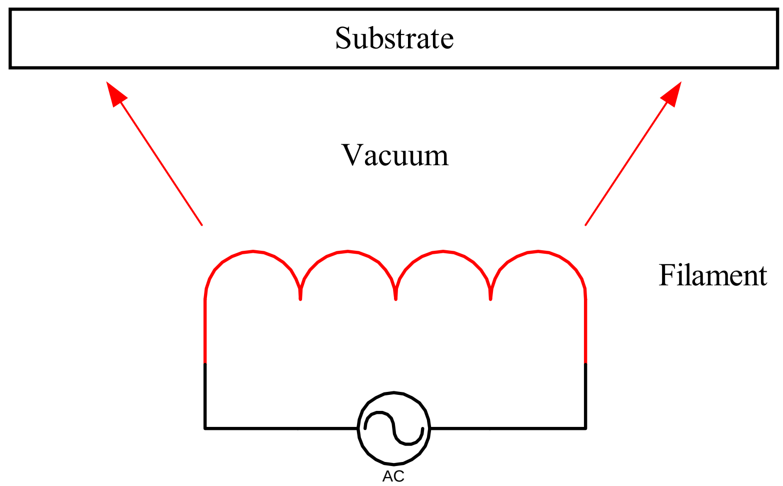


Figure 3. Vacuum evaporation (Society of Vacuum Coaters 1998).

Sputter Deposition.

In sputter deposition, the vapor is produced by momentum transfer and not from heating of the depositant material. High-energy ions from either a plasma or an ion gun are directed at a sputter target. The target contains the depositant material. When using a plasma, the sputter target is negatively charged to attract the positively charged plasma gas ions. Argon is a commonly used plasma gas for this purpose. The impact of the ions forces atoms to be displaced from the target. The displaced atoms are neutrally charged and have higher energies, 1 – 100 eV, than from a thermal process. The displaced atoms travel to the substrate. Figure 4 shows a simple sputter deposition arrangement.

The higher energy and scattering of the energetic particles provide better adhesion and coverage than with the low energy, line of sight, vacuum evaporation process. The deposition rates are lower than with vacuum evaporation (Bunshah 1994), and film properties are dependent on the angle of incident of the depositant (Society of Vacuum Coaters 1998). Ion plating was developed to overcome some of the drawbacks associated with both vacuum evaporation and sputter deposition (White 1995).

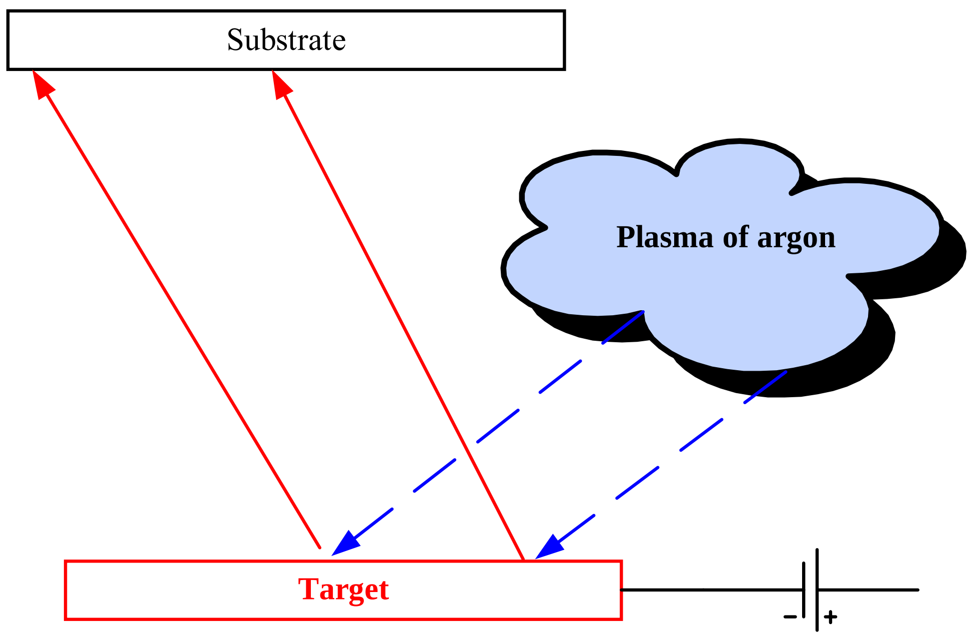


Figure 4. Sputtering (Society of Vacuum Coaters 1998).

Ion Plating

Ion plating is a deposition process in which high-energy ions are used to bombard the substrate during the deposition process. The depositant atoms may be vaporized using a thermal source as in vacuum evaporation or using a sputter target. The bombarding ions can be from either a plasma or an ion gun (known as ion beam assisted deposition, IBAD). In the case of a plasma, an inert gas such as argon is commonly used. The plasma is usually weakly ionized using a DC field, a radio frequency field (rf), or a microwave field. Only a small percentage of the gas is excited into an ionic state. The substrate is negatively charged using either a DC bias or radio frequency field. Due to the difference in electrical potential, the bombarding ions are accelerated toward the target. Figure 5 shows a simple ion plating arrangement.

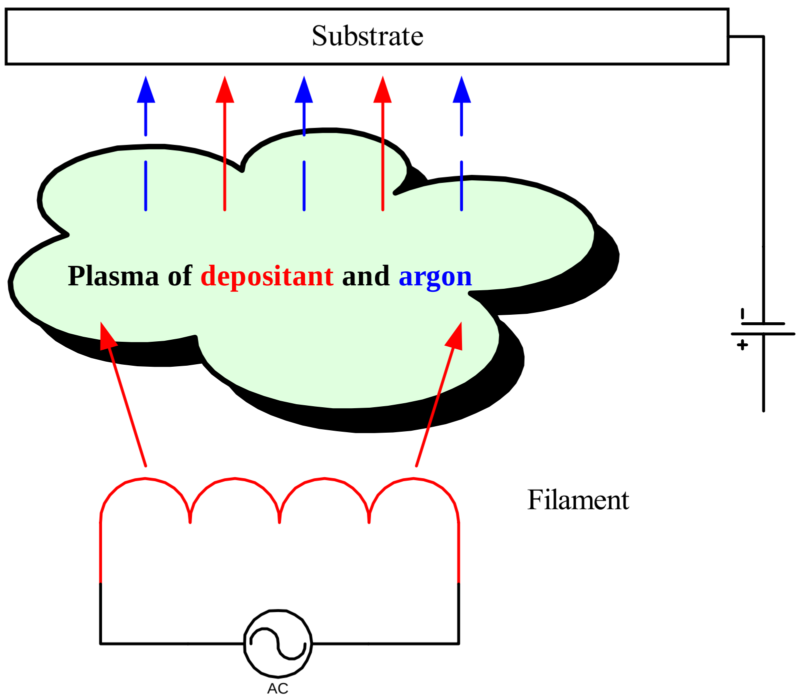


Figure 5. Ion Plating (Society of Vacuum Coaters 1998).

The energy of the bombarding ions should be great enough to disrupt the tendency of the film to form a columnar structure. The "atomic peening" of the film densifies the film and improves many of the film properties such as adhesion, adatom nucleation, interface formation, film growth, film morphology, electrical properties, optical properties, mechanical properties, and crystallographic orientation (Mattox 1994). In comparing adhesion between evaporated, sputtered, and ion plated films, Dini concluded that the better adherence of ion plated films was due, in part, to the higher energy of the depositant ions, which he found to be on the order of 50 -100 eV (Dini 1997). Dini also found evaporated atoms to have energy on the order of 0.1 - 0.2 eV and sputtered atoms to have energy of about 1-10 eV. Due to gas scattering and some surface effects, ion plating generally results in better surface coverage than vacuum evaporation or sputter deposition (Society of Vacuum Coaters 1998). Another benefit of ion plating is the ability to use the plasma to clean the surface of the substrate before the introduction of the depositant. This "back sputtering" removes surface contaminants that could interfere with the coating process (Mattox 1994).

In the case of ion plating using argon, the bombarding ions should have energies between 50eV and 300eV. The bombarding ions should add at least an additional 20 eV of energy per depositant atom to disrupt the columnar film growth. For example, if there were 10 depositant atoms for every bombarding ion, each ion would need an average energy of 200 eV. Bombarding ion energies below 50eV do not provide enough momentum to modify the film properties. Ion energies above 300eV result in excessive amounts of argon incorporated into the film which can cause voids and microporosity (Society of Vacuum Coaters 1998).

Regardless of the energy level, some gas atoms will be entrapped in the film. Depositant atoms can aggregate in the gas cloud. These aggregates form a sooty substance that contaminates the vacuum chamber and internal components. (White 1995).

PlasmaBond®

PlasmaBond® is the registered trademark of the vacuum deposition process developed by TXU and marketed commercially. PlasmaBond® represents improvements to ion plating technology. In this process, the depositant, which is usually a metal, is evaporated in a vacuum chamber. The depositant atoms become ionized by interaction with resonant photons from a radio frequency (rf) field established at the substrate. The rf field also has the effect of building a negative charge on nonconductors present in the field. Positively charged depositant atoms can then be accelerated toward a nonconducting substrate. Figure 6 shows a simplified PlasmaBond® ion plating arrangement (White 1995).

There are several advantages to the PlasmaBond® process. First, this process can be used on both conducting and nonconducting substrates. Second, the plasma developed is composed of only depositant material. Third, the depositant ions are imparted with enough energy, on the order of 100 eV, to penetrate the substrate material and embed themselves thus being mechanically anchored (Angelo 1994; Hopkins, Black, and Harrington 1997). Finally, substrate temperatures using PlasmaBond® typically remain below 125° F (Hopkins, Black, and Harrington 1997).

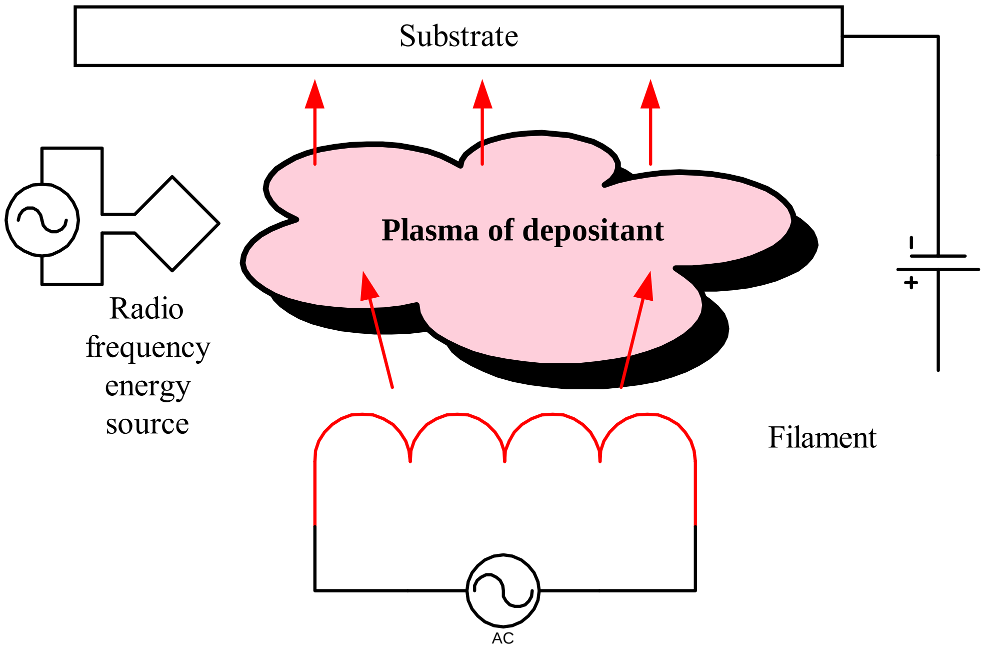


Figure 6. PlasmaBond® (White 1995).

PlasmaBond® Related Research

The following sections discuss the recent PlasmaBond® developments at TXU and research related to treatment of elastomeric stuffing box material by the Huber Corporation. The Huber Corporation used a gas-less ion plating process, which in this respect, is similar to the PlasmaBond® process. The review of literature did not reveal any other research in the area of coating of elastomeric seals using vapor deposition technology.

PlasmaBond® Anti-Galling Developments

TXU began investigating the use of PlasmaBond® in 1991. TXU's interest in this process was driven primarily by the desire to minimize delays in maintenance and damage to equipment associated with galling of metal components. This process was first used at TXU's fossil plants. Recent developments have centered on uses at TXU's nuclear power plant. Because of the sensitivity associated with new technology at a nuclear power plant, the following testing was used to justify the use of PlasmaBond® at TXU's Comanche Peak Steam Electric Station nuclear power plant.

TXU tested ¾ inch diameter carbon steel bolts that were exposed to 625° F for one month. Testing demonstrated that the treated surfaces resisted galling. TXU also conducted extensive testing on treated 7-inch diameter reactor vessel closure studs. Testing indicated that there was no significant wear with unlubricated studs after repeated removal cycles. In addition, there was no indication of galling. Subsequently, all of the reactor vessel studs (54 in each of the two vessels) have been treated and no galling has been detected. TXU had previously experience problems with reactor vessel studs binding and galling. TXU evaluated PlasmaBond® treated carbon steel fasteners repeatedly torqued to maximum service levels used in a typical pipe flange joint. There was no indication of galling after at least three assembly cycles (Harrington and Kidd 1998).

Stuffing Box Treatment

Angelo (Angelo 1994) performed laboratory tests of sulfur cured nitrile stuffing box material used in oil field equipment. The testing involved pressurizing an inverted cone stuffing box seal with nitrogen. A polished rod was continuously reciprocated at a rate of 20 strokes a minute through the seal. Leakage measurements were performed by capturing the nitrogen in a water trap and measuring the displacement of water in a graduated cylinder. One packing coated with a metal using a gas-less ion plating process and one uncoated seal were tested. Laboratory tests showed that the treatment significantly reduced the drag on polished push rods with packing seals. The laboratory test also showed that the treatment increased the time to leakage from 10 hours to 114 hours compared to the untreated packing. Angelo also analyzed data from both Huber Corporation and Arco Corporation field tests. Field tests at Arco compared coated, peroxide cured nitrile packing with uncoated, sulfur cured nitrile packing in heavy oil operations in California. In less than 200° F service, the coated packing did not last as long as the uncoated packing, 73 days compared to 86 days. However, the coated packing required less maintenance (fewer packing adjustments to reduce leakage). In service conditions greater than 200° F, the coated packing lasted significantly longer, 118 days as compared to 48 days. Normally, as temperature increases, the life of nitrile packing decreases in this type of service. The coated packing, however, tended to have an increased life as temperature increased. In the Huber field tests, the life of the packing also appeared to be increased using the treatment. This field testing was not as long or as extensive as the Arco testing and, therefore, not as conclusive. Noise pollution, which was a major irritant for nearby residents, was also evaluated in the Huber field testing. In this particular service, the oil well pumps ran dry for a significant portion of the cycle. The packing received little lubrication which resulted in squeaking noises from the stuffing box. Using a variety of different packing compounds, only 2-7 days of noise free operation had been previously achieved. The coated packing was able to eliminate the squeaking noise for 30 days.

CHAPTER 3

METHODS AND MATERIALS

The review of literature in chapter 2 demonstrated that there is a need to reduce and maintain low valve stem leakage. The review further indicated that, in some valves, elastomeric O-rings are used as valve stem seals. Lubrication is important during installation and operation of O-rings for them to insure proper sealing. A vacuum coating technology developed by TXU provides such a lubricant. This research focused on determining if the use of TXU's vacuum coating technology, PlasmaBond®, on elastomeric O-rings can reduce valve stem leakage.

The approached used in this research was to select a test valve that uses elastomeric seals and is representative of valves used in gas or volatile liquid service. Spare stem seals were obtained from the valve manufacturer and half were coated with a thin metal film using TXU's PlasmaBond® process. Each seal was installed in the test valve using the manufacturer's recommendation with the exception that half of the coated group and half of the uncoated group were installed without lubrication. Valve stem seal leakage was measured at the valve stem/bearing housing interface using a sniffing method.

Research Design

This research used a posttest-only experimental design with an untreated control group. No pre-testing was conducted because the seals could not be tested in the valve before the treatment without potentially affecting the results and because of the assumption that the seals procured for this testing are representative of the population of replacement seals (White 1987). By designing this research such that half of each group was installed with lubrication and half without lubrication, a comparison of the lubrication to the PlasmaBond® treatment with respect to valve stem leakage was made. In addition, the design of this research enabled a determination of whether these two factors, PlasmaBond® and lubrication, interact. The use of two factors in this manner was accommodated by a two level replicated complete factorial design (Diamond 1989). This research was designed to control those variables that could be controlled and could affect stem leakage. Variables that were not expected to affect the results or were uncontrollable (intervening variable) were not controlled.

Controlled Variables

The independent variable or factor that was the primary interest of this research was the presence of the PlasmaBond® coating. This coating was applied at TXU's PlasmaBond® facility using personnel and procedures developed under that facility's quality assurance plan. The O-rings to be coated were randomly selected from the O-rings purchased for this testing. The coated O-rings were physically segregated from the control group. In addition, the coated O-rings have a bright gold finish and it was virtually impossible to confuse them with the uncoated group (figure 7).

Figure 7. Coated O-ring on the left and uncoated O-ring on the right.

Lubrication was the other factor that was of primary interest in this research and was, therefore, the other independent variable or factor. The manufacturer recommends that valve stem seal O-rings be installed with Marfak #3 heavy grease or equivalent grease (Grove Valve 1980). TXU stocks Texaco Premium RB NLGI grade 2 grease as a standard lubricant. Dresser Industries confirmed that this is an equivalent grease suitable for this valve (Uhernick 2000). Half of each group was lubricated with the Texaco grease during installation. The lubricant used for this research was obtained from a single container of TXU warehouse stock grease. Lubrication was removed from the gland plate before installation of unlubricated O-rings.

The size and type of elastomeric O-ring were the moderating variables associated with the O-ring itself. As shown in equation 7, the amount of leakage through the O-ring is dependent on these characteristics of the O-ring. This valve uses Viton® as the standard material for the O-ring seal, however, other materials can be ordered. Viton® is very versatile and has a wide range of material resistance (Martini 1984; Parker Hannifin 1998). In addition, this material has low gas permeability and is suitable for vacuum processes (Parker Hannifin 1999). O-rings procured for this testing were specified to be the standard material and ordered by part number. The packaging material for these O-rings identified them as Viton®. In addition, one O-ring was destructively tested in the TXU's Overview test lab and confirmed to be Viton®. This O-ring comes in only one size and each O-ring was installed in the gland plate without any apparent difference in the O-ring size.

Type and thickness of the coating were the moderating variables associated with the PlasmaBond® process that could affect the leak rate of the test group O-rings (Parker Hannifin 1999). To control these variables, all of the O-rings were processed at the same time using the same equipment and technician.

Temperature, pressure, and type of test gas were the moderating variables associated with the test fluid that could affect the leakage through the O-ring (equation 7). Conducting the testing in an air-conditioned test area controlled the temperature and temperature was monitored at the bearing housing/valve stem interface during testing. Pressure was controlled during the testing through the use of a pressure regulator and pressure was measured using a precision pressure guage. High purity chromatography grade helium provided control of the test gas. All of the gas used during this research came from the same gas cylinder.

The fit, finish, material, and squeeze were the moderating variables associated with the valve gland that could affect leakage (Martini 1984; Parker Hannifin 1999). Use of a single test valve assured that gland fit, finish, and material were controlled. Squeeze was controlled by following manufacturer's recommended assembly practices which included bearing housing cap screw torque specifications (Grove Valve 1980). All of the tests were conducted with the cap screws torqued to the same value.

Valve cycle rate, valve position, and pressure hold time were the extraneous variables that were controlled during the testing. The manufacturer recommended that the valve be cycled at a rate of no more than 1 cycle per second due to valve seat considerations. The manufacturer indicated that a cycle rate of 10 to 15 cycles a minute should have no affect on the valve. During the testing the valve was operated manually and the cycle rate was controlled to not exceed a rate of 10 cycles per minute. The manufacturer also recommended that the valve be position from 1/8 to 7/8 open to assure that the valve stem is pressurized with the test gas (Uhernick 2000). During the testing, the valve was positioned to approximately half open during the leak measurements. After initial pressurization of each seal, pressure was allowed to stabilize for 20 minutes prior to taking any measurements (Leefe and Davies 1995). This allowed the fluid pressure to seat the O-ring in the gland.

Uncontrolled Variables

The extraneous variables that were not expected to confound the results and the intervening variable were not controlled in this research. The rate of pressurization was an extraneous variable that was not expected to influence the results of the testing so it was not specifically controlled. However, each test sequence was pressurized in a similar manner. Pressure was raised in a deliberate fashion due to laboratory safety concerns. Air flow around the test valve was not expected to confound the results because of the use of the sniffing method of leak detection (United States Environmental Protection Agency 1995) and because the testing was conducted in an air conditioned laboratory environment. Therefore, no specific controls were placed on air flow around the test valve.

Wear of the valve parts was the intervening variable that could have affected the results of the testing (Parker Hannifin 1999). Because only one valve was used throughout this research, the later tests could be affected more by wear than earlier tests. The affect of this variable was minimized by the use of multiple samples and by randomizing the test sequence (White 1987).

Sample Size

The determination of sample size followed the approach described by Diamond (1989). In this approach, the alpha and beta risks were first chosen. The necessary improvement or change to accept that there is a difference at those risk levels was chosen next. The sample size was then calculated for a normal distribution as a first approximation. The sample size for a student t distribution was calculated based on the preliminary normal distribution sample size. The actual sample sized was selected based on the Hadamard Matrix with a sample size greater than or equal to the t distribution sample size.

Risk and Improvement

In this research the consequence of committing a type I error, rejecting H_o when H_o is true, was that it would have been claimed that the new process improved leakage when, in fact, it did not. Ultimately, this could result in more expense incurred in spare seals. On the other hand, the consequence of committing a type II error, failing to reject H_o when, in fact, H_o is false, is that it would have been claimed that the new process makes no difference when, in fact, there was an improvement. This would have resulted in a missed opportunity to reduce maintenance cost associated with leak detection and repair. Because the consequences of committing both types of errors were similar, the significance of both errors were treated the same in this research.

The probability of committing a type I error, α, is generally chosen to be in the range of .01 to .1. It is a common practice to choose α to be .05 when there is no significant difference between type I and type II errors (Kvanli, Guynes, and Pavur 1996). For this research, α was chosen to be .05. Likewise, the probability of committing a type II error, β, was chosen to be .05.

The minimum amount of required improvement is the other factor necessary to determine the sample size. A larger amount of improvement will result in a smaller sample size at the same risks. This reduces the cost of the testing. In addition, there should be a distinct difference between the treated and untreated seals to justify the additional cost of treating the seals. I could find no existing data on the leakage through the seals for the test valve used for this research. Therefore, improvement as a function of population variance was chosen as the approach used to deal with improvement for determining sample size and objective criteria (Diamond 1989). An improvement, δ, of 2 standard deviations, 2σ, was chosen because this would represent a level outside approximately 95% of the population of the untreated seals assuming a normal distribution.

Normal Distribution Approximation

A normal distribution was assumed (figure 8) as a first approximation. A common assumption is that the variances of the treated and untreated populations are equal because this greatly simplifies the statistics. In addition, the problem of unequal variances virtually disappears when the sample sizes are large (>8) (Diamond 1989). In determining the sample size for this research, the variances of the populations of coated and uncoated seals were assumed to be equal and unknown. For this case, the sample size for a normal distribution at a specified α, β, and δ is given by equation 8 (Diamond 1989):

$$N = 2\left(Z_{\alpha} + Z_{\beta}\right)^{2} \frac{o^{2}}{\delta^{2}} \tag{8}$$

For a single sided test with the chosen α and β, $Z_{\alpha} = Z_{\beta} = 1.645$. This results in a normal distribution sample size of $N = 5.412$.

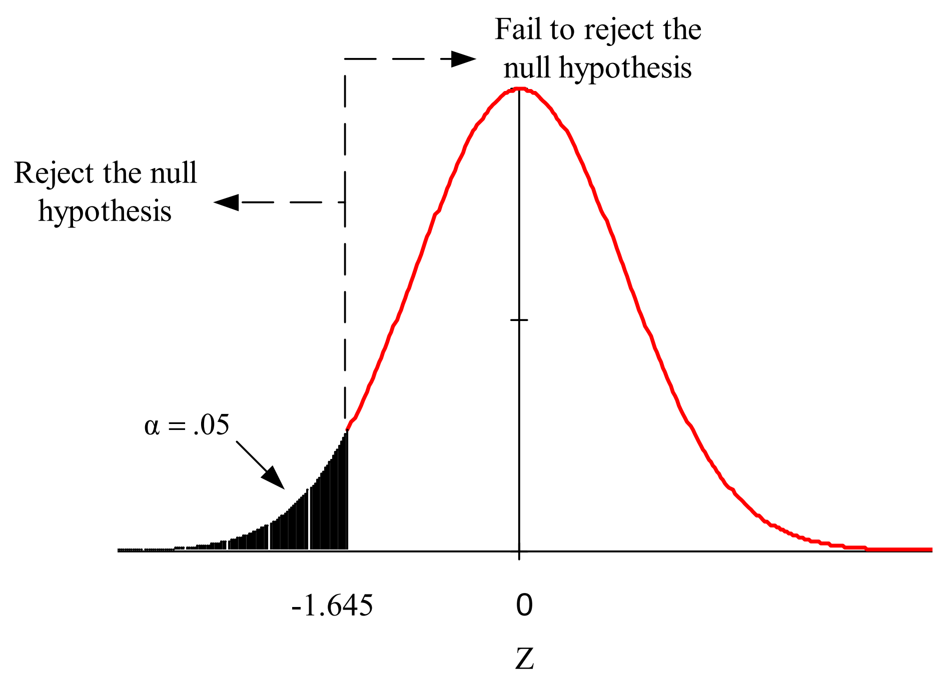


Figure 8. Normal distribution one tail test plotted using MathCad.

Student *t* Distribution Sample Size

A Student *t* distribution should be used to estimate the sample size and the objective criteria when the variance of a population is unknown and the estimated sample size is less than 30 (Kvanli, Guynes, and Pavur 1996). The use of a student t distribution requires that the number of degrees of freedom be specified. Use of a Hadamard matrix as discussed by Diamond (Diamond 1989), provided a means to design a two level, multivariable experiment and determine the sample size. The normal distribution sample size approximation corresponded to a 16 x16 Hadamard matrix (figure 9) where $N_{treated}$ = $N_{untreated}$ = 8. Because there were only two variables of interest (PlasmaBond® - A, grease –B), there were 12 Hadamard matrix columns (3, 4, 6-15) available to estimate the variance. Therefore, the degrees of freedom, ϕ, were 12. The sample size for the t-distribution is given by equation 9 (Diamond 1989):

$$N_{treated} = N_{untreated} = 2\left(t_{\alpha,12} + t_{\beta,12}\right)^2 \frac{\sigma^2}{\delta^2} \tag{9}$$

For a single sided test with the associated values of α, β, and ϕ, $t_{\alpha,12} = t_{\beta,12} = 1.79$. This resulted in t distribution sample sizes of $N_{treated} = N_{untreated} = 6.4$. Because the sample size using the t-distribution was > 4, and $\leq$ 8, the 16x16 Hadamard matrix was the correct size to use (Diamond 1989) and the corresponding sample size for this research was 8.

	0	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
1	1	1	-1	-1	-1	1	-1	-1	1	1	-1	1	-1	1	1	1
2	1	1	1	-1	-1	-1	1	-1	-1	1	1	-1	1	-1	1	1
3	1	1	1	1	-1	-1	-1	1	-1	-1	1	1	-1	1	-1	1
4	1	1	1	1	1	-1	-1	-1	1	-1	-1	1	1	-1	1	-1
5	1	-1	1	1	1	1	-1	-1	-1	1	-1	-1	1	1	-1	1
6	1	1	-1	1	1	1	1	-1	-1	-1	1	-1	-1	1	1	-1
7	1	-1	1	-1	1	1	1	1	-1	-1	-1	1	-1	-1	1	1
8	1	1	-1	1	-1	1	1	1	1	-1	-1	-1	1	-1	-1	1
9	1	1	1	-1	1	-1	1	1	1	1	-1	-1	-1	1	-1	-1
10	1	-1	1	1	-1	1	-1	1	1	1	1	-1	-1	-1	1	-1
11	1	-1	-1	1	1	-1	1	-1	1	1	1	1	-1	-1	-1	1
12	1	1	-1	-1	1	1	-1	1	-1	1	1	1	1	-1	-1	-1
13	1	-1	1	-1	-1	1	1	-1	1	-1	1	1	1	1	-1	-1
14	1	-1	-1	1	-1	-1	1	1	-1	1	-1	1	1	1	1	-1
15	1	-1	-1	-1	1	-1	-1	1	1	-1	1	-1	1	1	1	1
16	1	-1	-1	-1	-1	-1	-1	-1	-1	-1	-1	-1	-1	-1	-1	-1
		A	**B**			**-AB**										

Figure 9. 16x16 Hadamard Matrix (Diamond 1989).

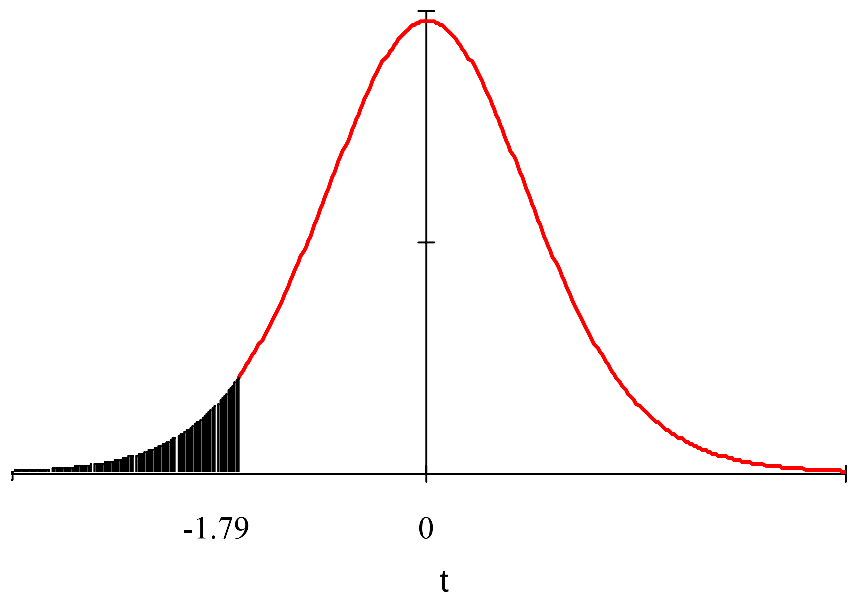


Figure 10. Student *t* distribution with 12 degrees of freedom, one tail test, plotted using MathCad.

Sample Means

The methodology for determining the difference in sample means using Hadamard matrices is discribed in detail by Diamond (1989). The difference between means of the samples that are treated with the PlasmaBond® process and those that are not is the sum of the trial results with the sign applied to each trial from column 1 of the Hadamard matrix divided by the sample size as shown in equation 10 (Diamond 1989):

$$\overline{X}_{PlasmaBond} - \overline{X}_{uncoated} = \frac{\sum A_{yes(+1)} - \sum A_{no(-1)}}{8} \tag{10}$$

Similarly, the difference between the means of the samples that are lubricated and those that are not is the sum of the trial results with the sign applied to each trial from column 2 of the Hadamard matrix divided by the sample size as shown in equation 11 (Diamond 1989):

$$\overline{X}_{greased} - \overline{X}_{ungreased} = \frac{\sum B_{yes(+1)} - \sum B_{no(-1)}}{8} \qquad (11)$$

Evaluating the difference between the samples when both variables are at the same level and those samples when the variables are at opposite levels can assess the interaction between the two factors. Column 5 of the Hadamard matrix is labeled "–AB". This indicates that it has the opposite sign of column 1 multiplied by column 2. The value in column 5 is negative when both variables are high or both are low, and the value is positive when the factors are at opposite levels. The interaction between the factors is the summation of the values in column 5. The average interaction can be expressed by equation 12 (Diamond 1989). This is simply the sum of the values in column 5 with the appropriate sign applied divided by the sample size (8).

$$\overline{X}_{+AB} - \overline{X}_{-AB} = \frac{\sum -AB(col5)_{+1} - \sum -AB(col5)_{-1}}{8} \qquad (12)$$

Variance

Columns 3, 4, and 6 through 15 of a 16 x 16 Hadamard matrix for a two factor experiment provide an estimate of the sample variance, S^2. The sum of the trial results from the respective column divided by the number of trials provides an estimate of the variance with one degree of freedom as shown in equation 13 (Diamond 1989).

$$S^2_{column} = \frac{\sum_{column} results}{16} \qquad (13)$$

The average variance with 12 degrees of freedom is the sum of the column variances divided by the number of columns (12) used to estimate the variance (equation 14) (Diamond 1989).

$$S^2_{Ave} = \frac{\sum S^2_{columns}}{12} \qquad (14)$$

Objective Criteria

The objective criteria provided the measures by which the results of the testing were compared with to determine if there were sufficient differences between the two populations to reject the null hypotheses. If the difference in the means was greater than the following test criterion, then the null hypothesis should have been rejected and the alternate hypothesis accepted. The objective criteria for the null hypotheses for research questions 1, 2, and 3 are provided in equations 15, 16, and 17 (Diamond 1989), respectively.

$$\left|\overline{X}_{PlasmaBond} - \overline{X}_{untreated}\right|^{*} = t_{\alpha} S_{Ave}\sqrt{\frac{1}{N}+\frac{1}{N}} \tag{15}$$

$$\left|\overline{X}_{greased} - \overline{X}_{ungreased}\right|^{*} = t_{\alpha} S_{Ave}\sqrt{\frac{1}{N}+\frac{1}{N}} \tag{16}$$

$$\left|\overline{X}_{+AB} - \overline{X}_{-AB}\right|^{*} = t_{\alpha} S_{Ave}\sqrt{\frac{1}{N}+\frac{1}{N}} \tag{17}$$

Test Sequence

The 16 x 16 Hadamard matrix with a sample size of 8 resulted in a replicated complete factorial design because each combination of the two factors was tested more than once (Diamond 1989). The actual test sequence was determined by drawing the trial number out of a hat. This randomized the sequence of the trials to minimize the effect of intervening variables and any unidentified extraneous variables. Table 2 shows the test sequence used.

Table 2. Trial versus test sequence.

Trial	PlasmaBond Factor A	Grease Factor B	Factor Combination	RandomTest Sequence
1	Yes	No	a-	7
2	Yes	Yes	ab	10
3	Yes	Yes	ab	4
4	Yes	Yes	ab	1
5	No	Yes	-b	6
6	Yes	No	a-	15
7	No	Yes	-b	8
8	Yes	No	a-	2
9	Yes	Yes	ab	9
10	No	Yes	-b	3
11	No	No	--	12
12	Yes	No	a-	5
13	No	Yes	-b	13
14	No	No	--	11
15	No	No	--	16
16	No	No	--	14
Total	8-Yes, 8-No	8-Yes, 8-No	4-a, 4-ab, 4-b, 4-neither	

Test Valve

TXU Pipeline Services provided me with a valve that is representative of valves used in gas storage and delivery systems. Natural gas storage and delivery service was chosen because many of the EPA limits are referenced to methane (U.S. Code of Federal Regulations Part 60: Standards of performance for new stationary sources 2000). In addition, valves used by TXU Gas Pipeline are industrial grade valves that are designed for volatile fluid or gas service. The valve provided was a new 2 inch, class 300, manual ball valve manufactured by the Grove Valve Division of Dresser Industries (figure 11). This particular valve was selected because of the high use of this size and type valve in TXU's gas pipeline and distribution systems (English 1999), and because of size and support system limitations at the testing laboratory. In addition, 2-inch, class 300 valves were used by some of the researchers found during the review of literature (Hoyes and Thorpe 1997; Lowe 1995).

Figure 11. Grove Valve in vertical position.

This valve has two elastomeric O-rings as valve stem seals (figure 12). The upper stem seal is redundant to the lower seal and allows for continued operation if for some reason the lower stem seal fails (Grove Valve 1999). There is also an O-ring used as a weather seal (figure 13). This O-ring prevents the intrusion of water and foreign material into the seal assembly and is not pressure retaining (Hammer 2000).

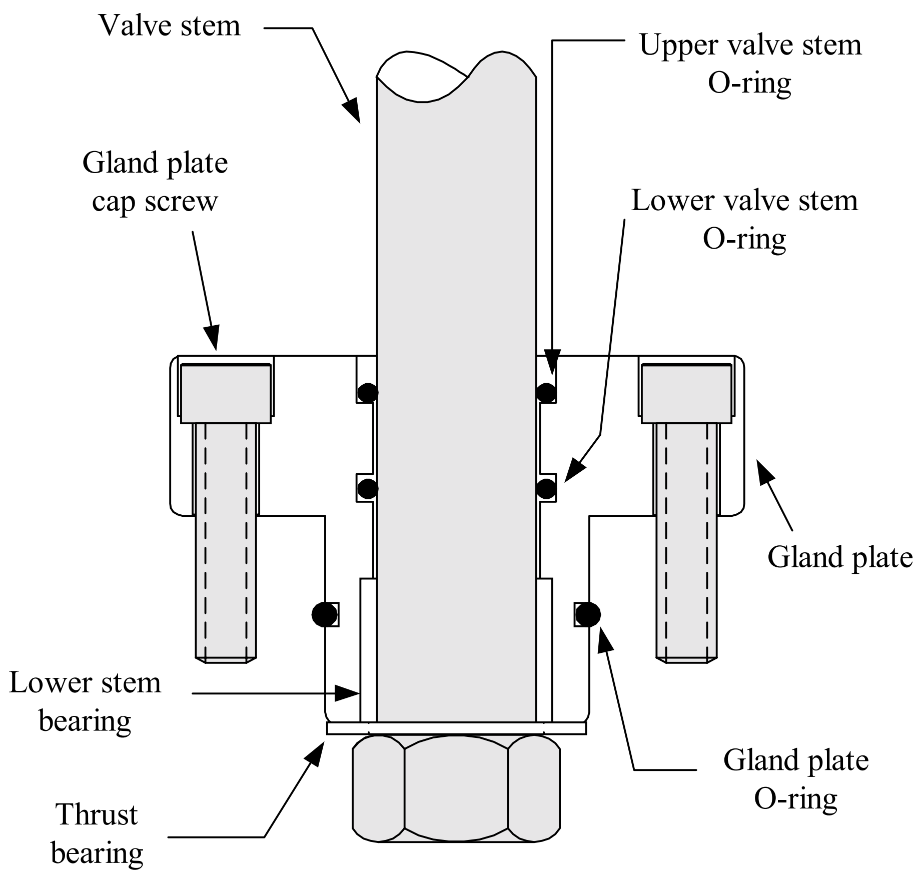


Figure 12. Gland plate cutaway.

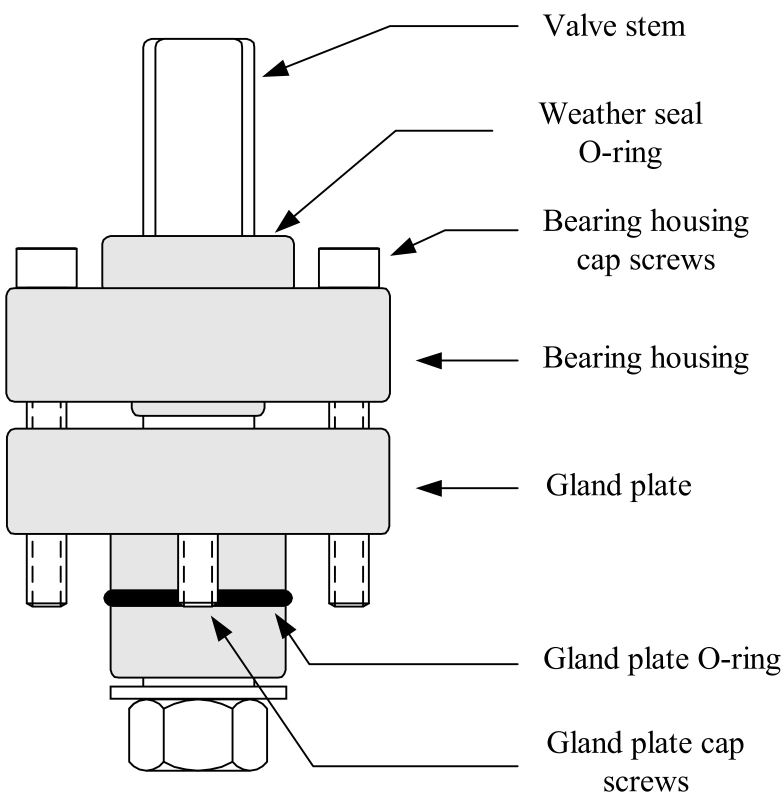


Figure 13. Grove valve stem and seal assemblies.

Leak Detectors

Leakage through an O-ring is a function of the diameter of the O-ring, gas permeability, differential pressure, Q factor, and squeeze as was shown in equation 7. Pressure, diameter, and gas permeability were fixed and known for this research. Squeeze and Q factor were not known, however, from table 1, Q factor ranges from 0.72 to 1.5 and squeeze ranges from .08 to .25 for dynamic seals (Parker Hannifin 1999). Using equation 7 discussed in chapter 2, the calculated expected leakage ranged from 2×10^{-5} to 6×10^{-5} cc/second. Note that Parker does caution that this formula is only a rough approximation and is subject to a lot of variability especially for testing using helium (Parker Hannifin 1999).

Two different leak detectors were used during this research. Both of these instruments are portable units that use a thermal conductivity difference from air to detect a leak. A GOW MAC 21-250 leak detector (figure 14) was used primarily for leak location. This detector has a sensitivity of 1 x 10^{-5} cc/sec for helium and has two ranges, 1X and 100X (Gow Mac 2000). This instrument indicates leakage on a meter with a 0-50 division scale. Five divisions or 10 % deflection on the low range indicates leak detection at the sensitivity limit of this instrument. The deflection of the meter is roughly proportional to the amount of leakage and, therefore, the low range corresponds roughly to a leakage range of 1 x 10^{-5} cc/sec to 1 x 10^{-4} cc/sec. This instrument has a very fast response time that is considered to be nearly instantaneous (Mathews 2000). Because of this instrument's fast response time, difficulty in correlating the meter readings to a leak rate, and lack of sufficient overlap between the upper and lower ranges, this instrument was used for initial leak localization and as a check of the other instrument which was used for leak quantification.

Figure 14. GOW MAC 21-250 leak detector.

An Ion Science Gas Check 3000IS was the other leak detector, and it was used primarily for quantification of leaks. The response time of this instrument is 1 second. This instrument provides a digital readout in cc/sec principally calibrated by the manufacturer for helium with a sensitivity of 1 x 10^{-5} cc/sec (Ion Science 2000). This minimum sensitivity roughly correlates to about 160 ppmv for helium (Matheson-Trigas 2000). The Gas Check 3000IS has auto ranging and peak hold features as well.

Figure 15. Ion Science Gas Check 3000IS.

Testing Methods and Procedures

Testing involved first obtaining sample seals and separating them into either the control or test group. The test group samples were then treated using the PlasmaBond® process. The test valve and associated test equipment were set-up at TXU's Procurement Overview Test facility and leak measurements were performed on the coated and uncoated seals.

Sample Selection

Valve stem seals were obtained from TXU Pipeline Services. These seals were ordered for this project and had not been stocked by TXU. The seals were all placed into a single container. The seals were then randomized and each seal was drawn individually. The seals were alternately placed in either the test group or control group.

Sample Preparation

The test group was coated using TXU's proprietary PlasmaBond® process. All test group O-rings were treated at the same time. The O-rings were suspended in a vacuum chamber using thin metal wires (figures 16 and 17). After the O-rings were positioned in the vacuum chamber, the chamber was evacuated and argon was introduced. An rf field was applied and ionization of the argon occurred. The argon was used to backsputter the surface of the O-rings. This cleaned the surfaces of oil and contaminants. The chamber was evacuated again to remove the argon. The O-rings were then ready for the coating process

Figure 16. Test group loaded into the vacuum chamber.

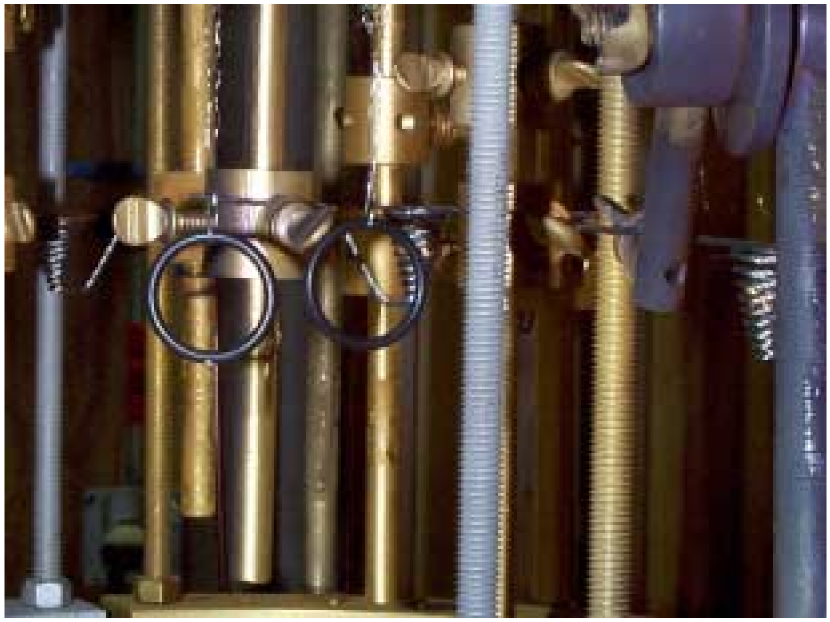


Figure 17. Close-up of test group before treatment.

The composition and thickness of the coating was provided by Hopkins (Hopkins 1999). Three different metal layers were applied. First, a film layer of a carbon stabilizing material layer that reacts with stray carbon atoms was deposited on the seals. This layer is composed of material that readily forms carbides such as iron, chromium, or titanium. Titanium was applied for this research because it is the standard base layer in this process. The first layer was applied by evaporating a crucible of titanium using a tungsten filament and by controlling the rf field to regulate the plating process. This first layer was deposited to a thickness of about 2000 angstroms.

A second barrier layer was applied to contain stray carbon atoms. In this process, this layer is optional but was used in this research. This layer is typically nickel because nickel carbide does not exist in a solid state. The nickel layer was deposited in a similar manner as the titanium layer. This layer was deposited to a thickness of about 1000 angstroms.

A final layer was applied over the stabilizing and barrier layers for lubrication. This layer is typically of material with low friction properties such as gold, silver, indium, palladium-silver, tin-silver, copper-nickel, brass, or bronze. For this research, gold was applied because it is chemically inert with sulfur. Sulfur is added as an odorant in gas pipelines and could react with the surface layer. The gold layer was applied in the same manner as the other two layers. The final layer was deposited to a thickness of about 3000 angstroms. The total thickness of the film layers was about 6,000 angstroms (about 40 millionths of an inch thick). The total amount of material deposited on each O-ring was approximately 4 mg. Figure 18 shows the vacuum chamber in the closed position during the coating process, and figure 19 shows the O-rings after the treatment.

Figure 18. Coating of the O-rings.

Figure 19. O-rings after coating.

Test Set-up

The test setup used in this research is similar to the testing performed by Leefe (Leefe and Davies 1995). Figure 20 shows the basic setup. Flanges were attached to each of the valve ports. One flange was a blind flange and the other was a blind flange that contained a tapped through hole. The valve was oriented in the vertical position to be able to fit into the valve test stand. In addition, the manufacturer's recommended practice for maintenance is that the valve be in this position. The valve was then placed in the test stand and the stand was secured.

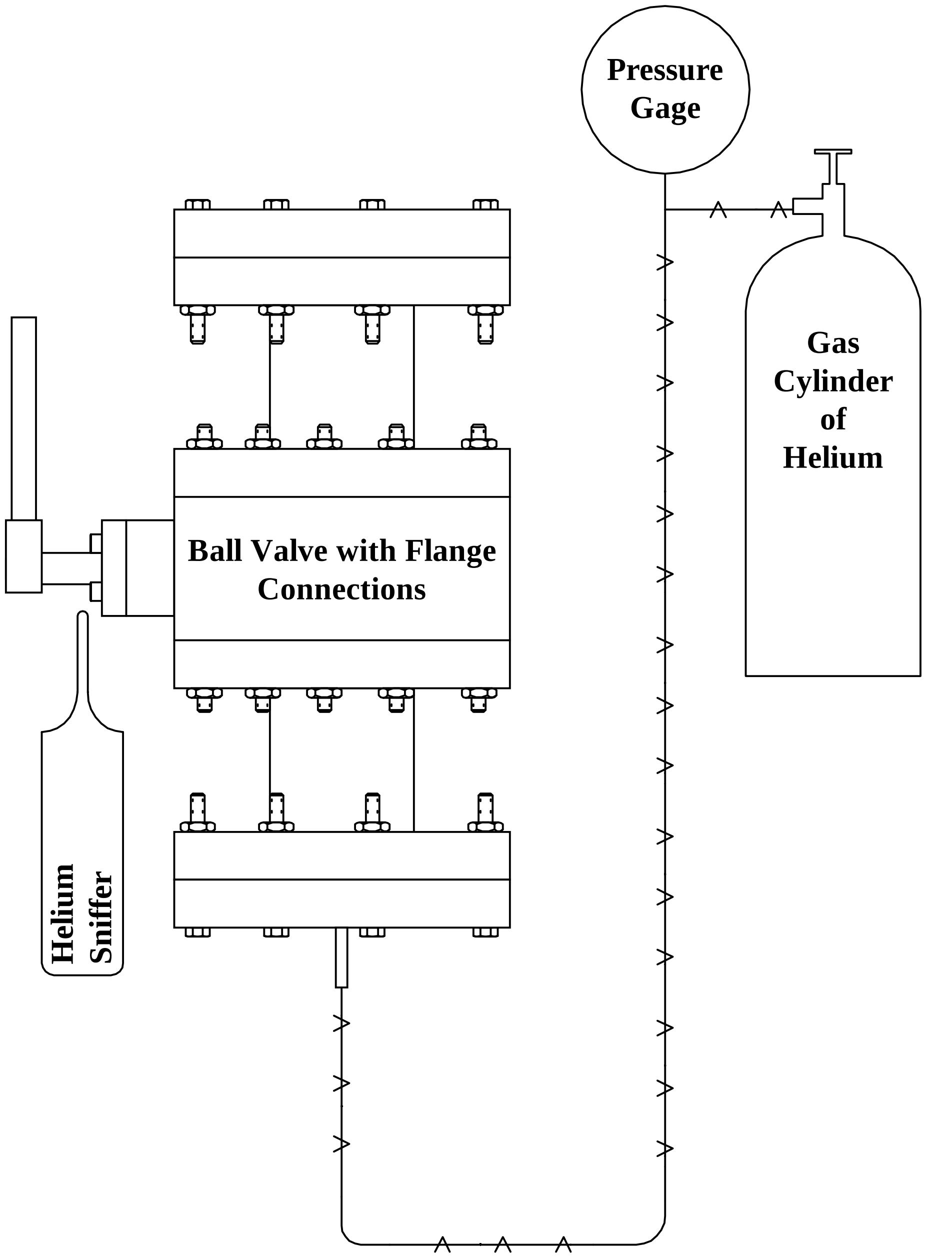


Figure 20. Test configuration.

The lower stem seal and weather seal were removed from the valve. This valve has redundant stem seals and is designed to function with only one (Grove Valve 1999). The removal of the lower seal simulates the failure of the lower seal and allowed testing of only one O-ring at a time. The weather seal does not provide a sealing function (Hammer 2000) and removal facilitated stem seal replacement and stem leakage measurement.

The helium supply was accomplished by connecting a high purity (99.995%), 2400 psi, helium gas cylinder to the tapped flange using ¼ inch stainless steel instrument tubing. Helium was chosen as the test gas because of laboratory safety considerations (Garrigues, Birembaut, and Ledauphin 1997; Leefe and Davies 1995) and because leakage was easily measured using the available portable leak detectors (Gow Mac 2000; Ion Science 2000). In addition, use of helium is common in conducting laboratory leak testing and can be roughly correlated to methane (Leefe and Davies 1995; Lowe 1995).

A gas regulator was attached to the gas cylinder, however, a calibrated, high precision pressure gage, 0-600 ± 3 psi, was used to monitor pressure. The test pressure was chosen to be 500 ± 25 psi because this pressure is typically the maximum working pressure used in TXU's pipeline and natural gas distribution systems for this type of valve (English 1999). This test pressure is within the 750 psi maximum working pressure for a class 300 valve at room temperature (American Society of Mechanical Engineers 1981; Uhernick 2000). In addition, this test pressure was within the range of pressures found during the review of literature, which were 7 bar (102 psi), 20 bar (290 psi), 40 bar (580 psi), and 50 bar (725 psi) (Harrison et al. 1995; Leefe and Davies 1995; Lowe 1995;

Ueda and Fujiwara 1997). Figure 21 shows the test valve installed in the test stand with the gas supplied from the gas cylinder.

Figure 21. Test valve in test stand.

<u>Test Procedure</u>

The test valve bearing housing, gland plate, upper stem seal and stem were removed prior to each test. Although the upper stem seal can be replaced without removing the stem, stem removal eased the removal of the upper stem seal that was used in the prior test. For test trials that were to be performed without lubrication, the gland plate upper stem seal area was cleaned of any residual lubricant before that test sequence. The stem and gland plate were reinstalled and the gland plate cap screws were torqued to

150 in-lbs using a calibrated breakaway torque wrench. The manufacturer's recommended torque range for cap screws on this size valve is 120 to 180 in-lbs (Grove Valve 1980). For test sequences that involved lubrication, the upper stem seal O-ring was then lubricated by liberally applying grease manually to the surface of the O-ring. The upper stem seal to be tested was installed by sliding it along the stem and seating it in the gland plate. The bearing housing was then installed and the bearing housing cap screws were torqued to 150 in-lbs using the breakaway torque wrench. The valve was then position to the half open position.

The valve was pressurized using the pressure regulator and the high precision pressure gage was used to monitor pressure during pressurization and testing. After the pressure was raised to 500 ± 25 psi, pressure was allowed to stabilize for 20 minutes (Leefe and Davies 1995) prior to cycling so that the O-ring would be seated by pressure into the gland plate.

The technique used for leak measurement followed EPA guidelines (United States Environmental Protection Agency 1995; United States Environmental Protection Agency 1999). Prior to any measurement, each detector was zeroed at least one meter away from the test valve. The tip of the each detector was placed directly at the valve stem/bearing housing interface. When leakage was found, the tip of a detector remained at the location of the leak until a maximum reading was obtained and for a duration of at least twice the response time. The GOW MAC 21-250 was used to slowly sweep the circumference of the interface to locate a leak. The Ion Science 3000IS was then used to quantify the leak.

The maximum reading of both instruments, pressure, and temperature at the bearing housing were recorded for each cycle measurement.

Figure 22. Locating the leaks using the GOW MAC 21-250.

Figure 23. Leak quantification using the Gas Check 3000IS.

Each test sequence consisted of a zero cycle measurement taken after the pressure stabilization period. The valve was then cycled, full open to full open, 10 times and leak measurements were taken with the valve in a half open position. In a similar fashion, the valve was then cycled 25, 50, and 100 times and measurements taken at each plateau. The test valve was cycled at a rate less than ten times a minute for any cycle test. The final plateau of one hundred cycles was chosen as the cycle limit for each test sequence because this number of cycles is within the typical range of manual on/off valve testing (Butcher 1997). After the conclusion of the 100 cycle measurements, the valve was positioned to the full open position and pressure was bled off.

CHAPTER 4

RESULTS AND ANALYSIS

Leak testing was conducted at TXU's Procurement Overview test facility. This facility was used exclusively for this research during the test periods. The results of the testing were recorded manually and later entered into a Microsoft® Excel spreadsheet. Test of the objective criterion, using the equations discussed in chapter 3, was the primary method employed to analyze the test data. Interaction between the two factors was also analyzed by plotting the leakage for each combination of the factors. Additionally, several observations in the area of the test sequence, leak location, and visual appearance of the valve parts were included as part of the analysis.

Tests of Objective Criteria

Tables 3, 4, and 5 are based on Hadamard matrices (appendix A) and show the results of the testing compared to the objective criteria. Each of these tables contains the average standard deviation, test criterion, and the difference in means between treated and untreated samples for each cycle plateau.

Table 3 corresponds to research question 1: Will the use of elastomeric O-rings with PlasmaBond® metallic coatings reduce stem leakage? The objective criteria were computed using equation 15. Table 3 shows that, except for the 50 cycle plateau, the testing resulted in a failure to reject the null hypothesis.

Table 3. PlasmaBond® test of null hypothesis

Cycles	Ave Standard Deviation	Test Criterion	Mean Coated – Mean Uncoated	Null Hypothesis
0	1.0×10^{-4}	-9.1×10^{-5}	-8×10^{-5}	Fail to reject
10	1.1×10^{-4}	-9.9×10^{-5}	-4×10^{-5}	Fail to reject
25	9.9×10^{-5}	-8.9×10^{-5}	-3×10^{-5}	Fail to reject
50	9.2×10^{-5}	-8.3×10^{-5}	-9×10^{-5}	Reject
100	1.2×10^{-4}	-1.1×10^{-4}	-1×10^{-5}	Fail to reject

Similarly, table 4 corresponds to research question 2: Will the use of a lubricant on elastomeric O-rings reduce stem leakage? The objective criteria were computed using equation 16. Table 4 shows that the testing resulted in a failure to reject the null hypothesis at each cycle plateau.

Table 4. Lubrication test of null hypothesis

Cycles	Ave Standard Deviation	Test Criterion	Mean Lubricated – Mean Unlubricated	Null Hypothesis
0	1.0×10^{-4}	-9.1×10^{-5}	-7×10^{-5}	Fail to reject
10	1.1×10^{-4}	-9.9×10^{-5}	-5×10^{-5}	Fail to reject
25	9.9×10^{-5}	-8.9×10^{-5}	-4×10^{-5}	Fail to reject
50	9.2×10^{-5}	-8.3×10^{-5}	-6×10^{-5}	Fail to reject
100	1.2×10^{-4}	-1.1×10^{-4}	-3×10^{-5}	Fail to reject

Table 5 corresponds to research question 3: How does the PlasmaBond® treatment along with the use of a lubricant affect sealing capability? The objective criteria were computed using equation 17. Table 5 shows that the testing resulted in a failure to reject the null hypothesis at each cycle plateau.

Table 5. Interaction of factors test of null hypothesis

Cycles	Ave Standard Deviation	Test Criterion	Mean High – Mean Low	Null Hypothesis
0	1.0×10^{-4}	-9.1×10^{-5}	-7×10^{-5}	Fail to reject
10	1.1×10^{-4}	-9.9×10^{-5}	-8×10^{-5}	Fail to reject
25	9.9×10^{-5}	-8.9×10^{-5}	-6×10^{-5}	Fail to reject
50	9.2×10^{-5}	-8.3×10^{-5}	-6×10^{-5}	Fail to reject
100	1.2×10^{-4}	-1.1×10^{-4}	-7×10^{-5}	Fail to reject

Interaction of the Factors

The results were analyzed based on factor interaction as discussed by Diamond (1989). Although the test of the hypothesis for interaction between the two factors failed to reject the null hypothesis, this analysis was performed to determine if any additional insight could be gained. The results from each combination of factors were averaged and plotted for each cycle plateau. For example, the trials in which the samples were uncoated and unlubricated, trials 11, 14, 15, and 16, were averaged and plotted as a single point.

Plots of the factor interactions for each of the cycle plateaus are shown in figures 24 through 28. These plots show that for lubricated O-rings, leakage at each cycle plateau can increase or decrease using the PlasmaBond® treatment. Unlubricated O-rings showed a consistent reduction in leakage using the PlasmaBond® treatment.

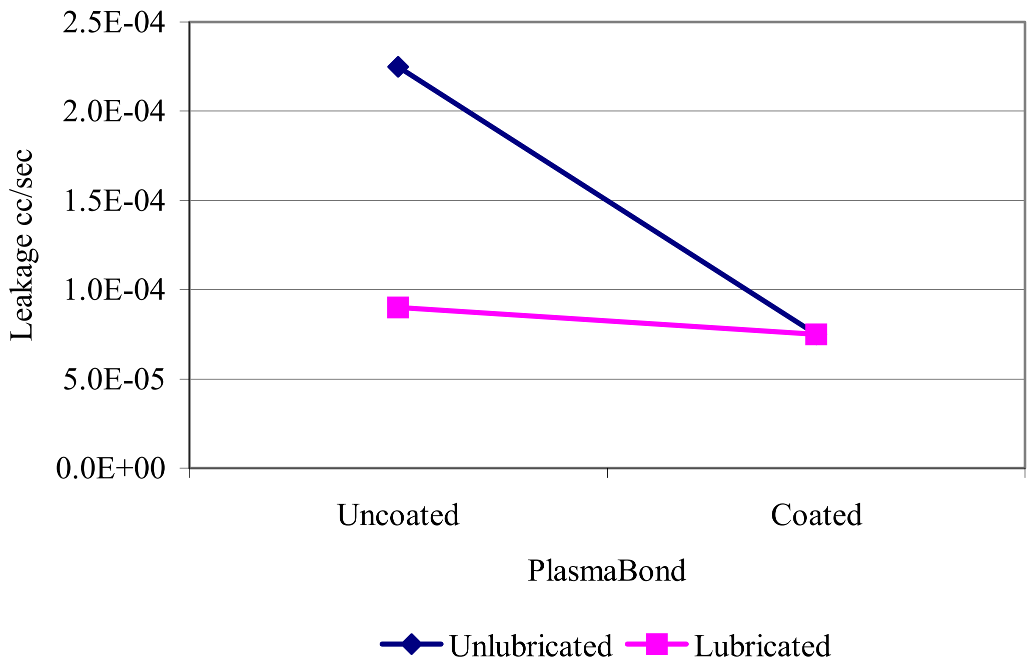


Figure 24. Plots of AB interaction at 0 cycles.

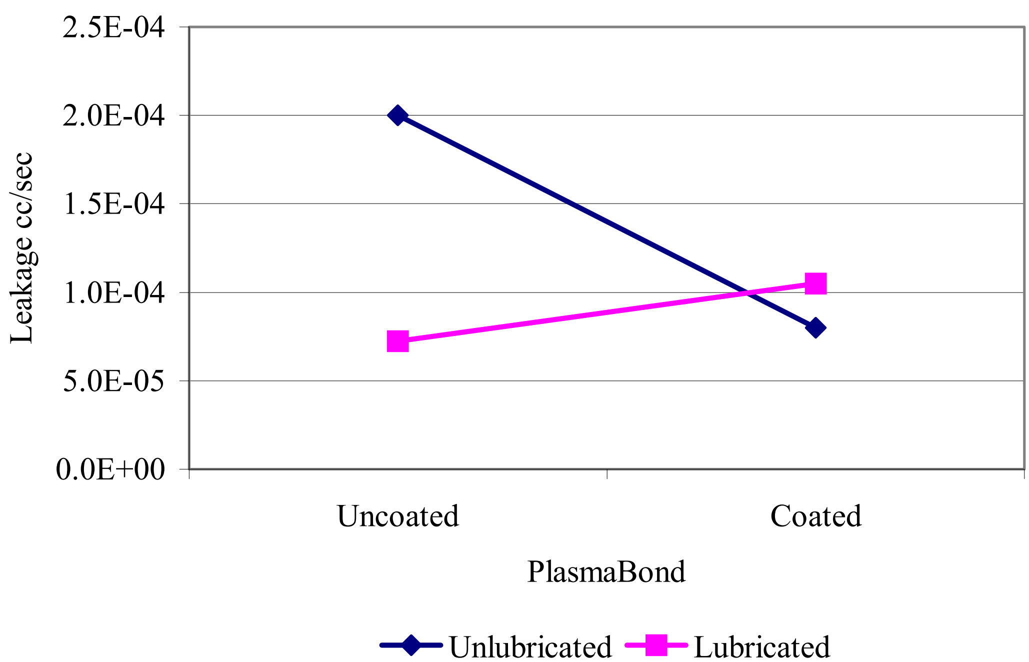


Figure 25. Plot of AB interaction at 10 cycles.

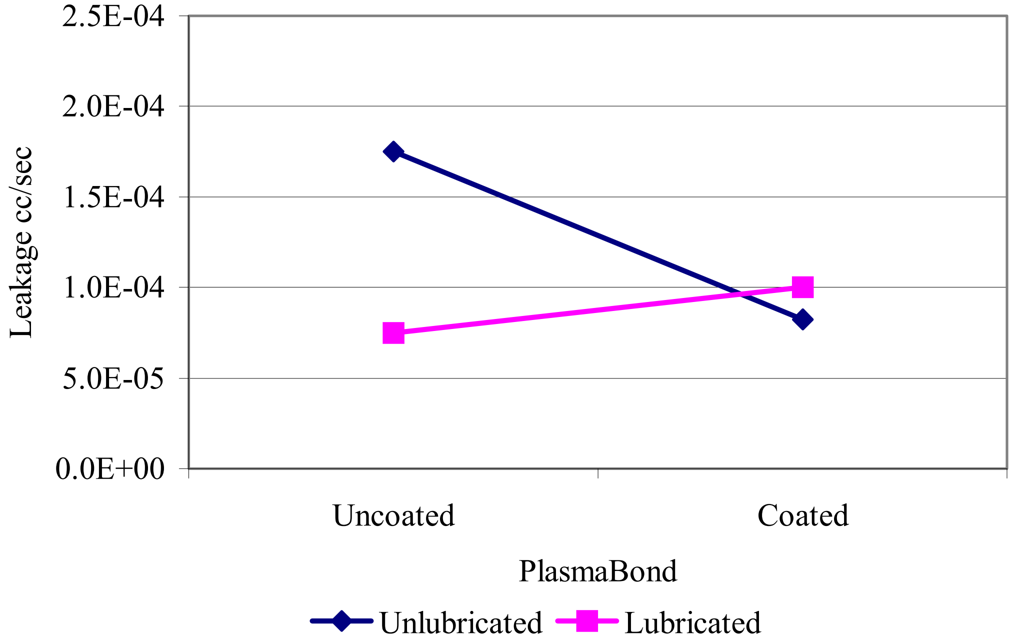


Figure 26. Plot of AB interaction at 25 cycles.

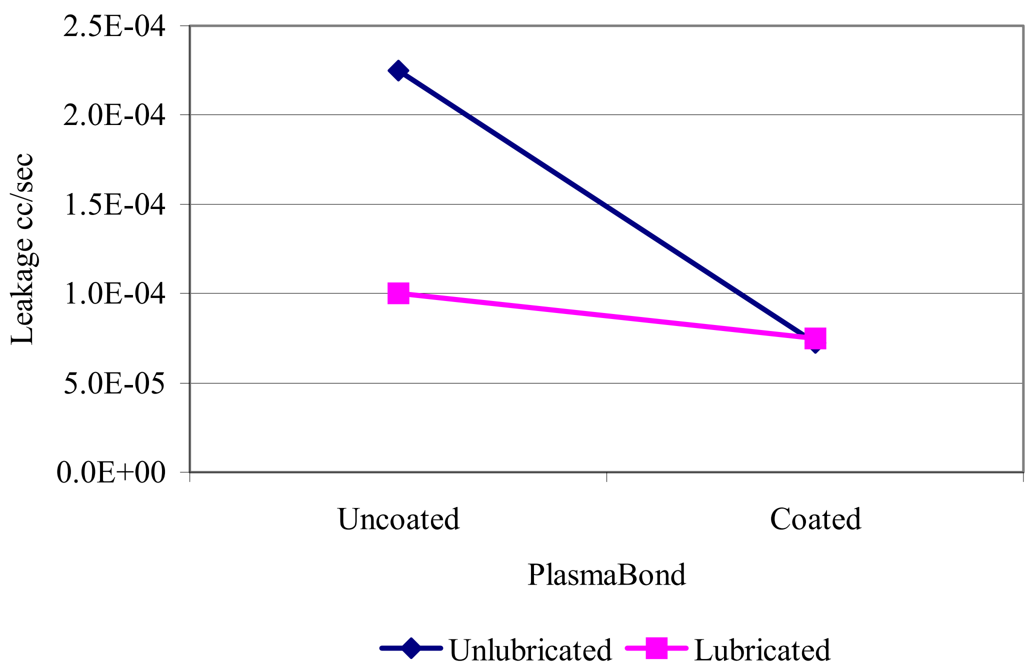


Figure 27. Plot of AB interaction at 50 cycles.

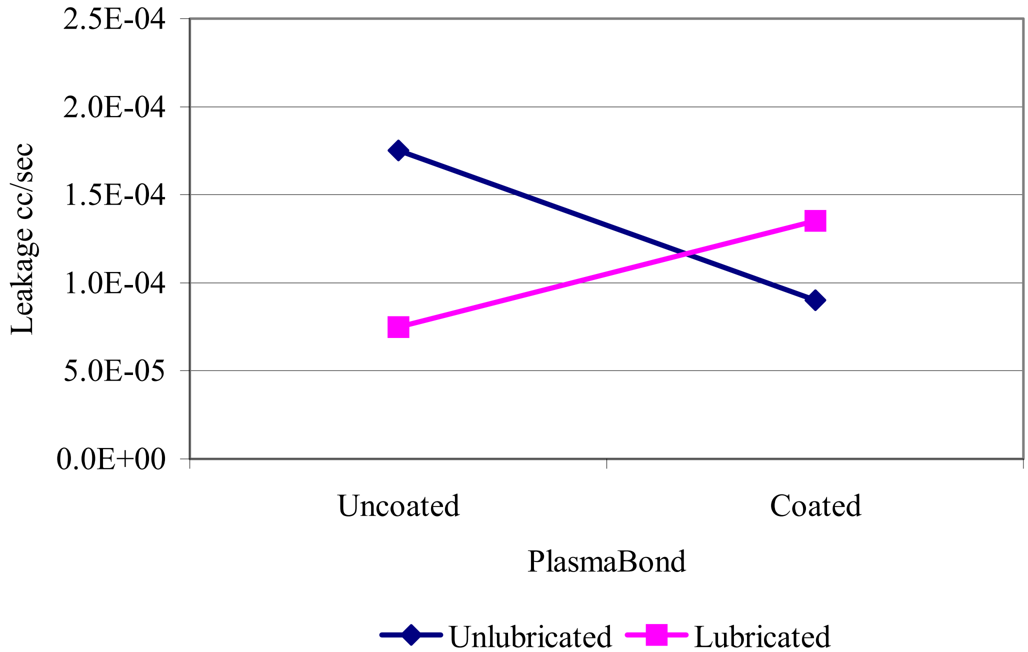


Figure 28. Plot of AB interaction at 100 cycles.

Observations

This research was primarily concerned with measuring leakage in accordance with the research design discussed in chapter 3. During the testing, however, observations were made in three areas that warrant discussion. These observations involved the test sequence; the leak location; and the visual appearance of the stem, gland plate, and coated O-rings.

Test Sequence Observations

The test sequence was determined by randomizing the order of the trials. This was done to minimize the affect of the intervening variable and any unidentified extraneous variables. It was noted during the testing that the leakage appeared to increase as the testing progressed. Figure 24 shows the average leak rate, including all of the cycle plateaus, for each test sequence.

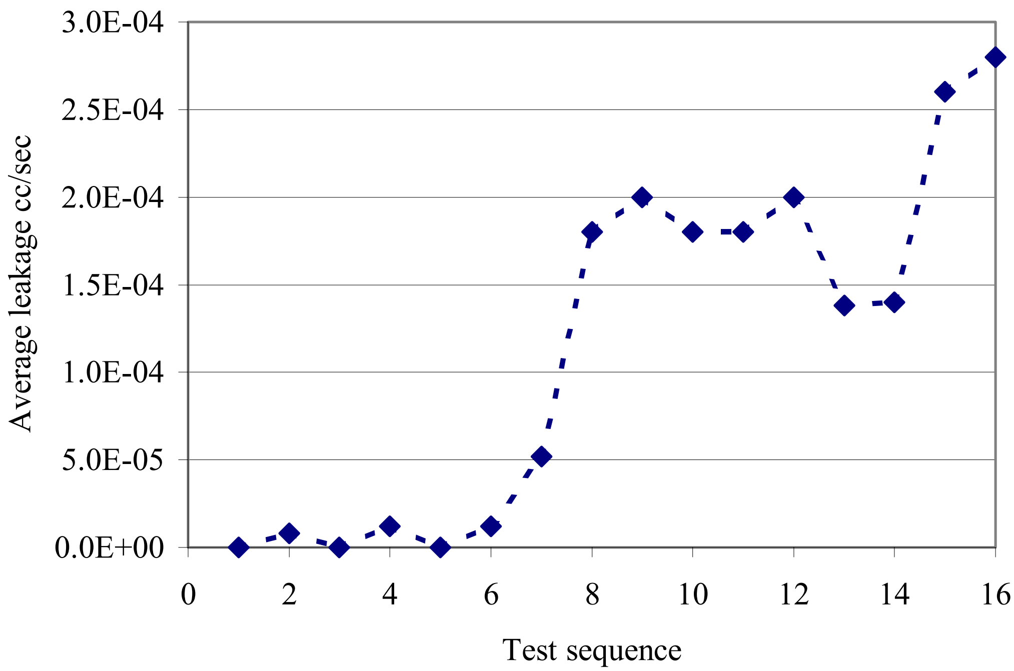


Figure 29. Plot of average leakage for each test sequence.

<u>Leak Location Observations</u>

A leak was usually detected initially using the GOW MAC 21-250 instrument. The Ion Science detector would then be placed at that location and the leak rate measured. Both the instruments used have an audio feature, which varies as the leak rate varies. Based on the sound, it was determined when the leak rate had reached its peak at a location. The leak rate at a location would increase over a period of time and then decrease. When sweeping the valve stem/bearing housing interface (figure 30) during each cycle test, the location of the leaks appeared to move.

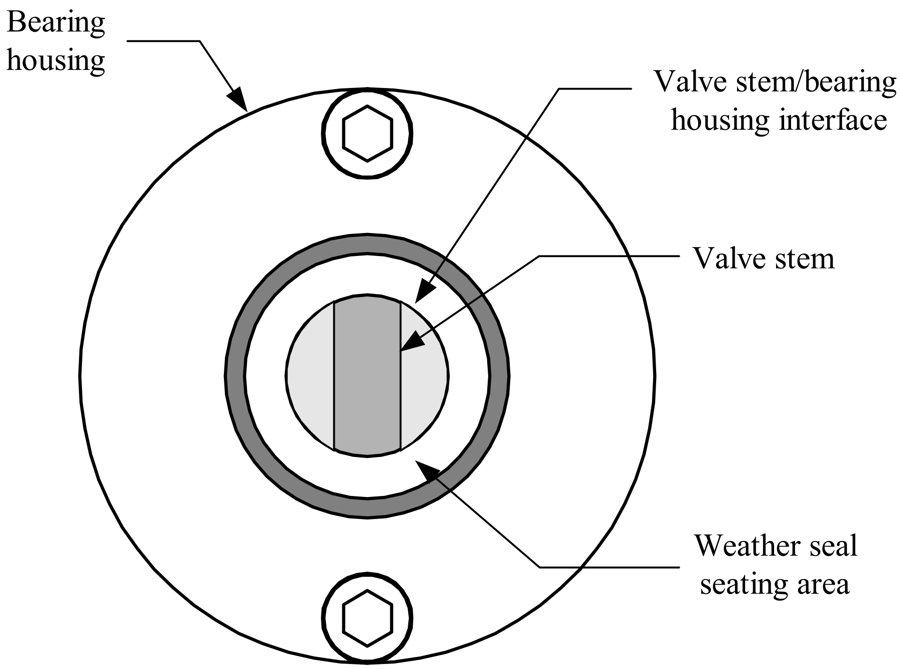


Figure 30. Valve stem/bearing housing interface.

Visual Appearance

The valve stem, valve gland plate, and O-rings were visually inspected with the naked eye after the conclusion of all of the testing. The valve stem showed a distinct gold band at the location of the upper O-ring seal (figure 31). The gland plate showed little if any transfer of material (figure 27). Neither of these parts displayed any obvious signs of wear. The O-rings treated with the PlasmaBond® process showed signs of coating removal (figure 28). There were two distinct parallel bands where the coating had been worn away. These bands appeared to be where the O-ring made contact with the mating parts.

Figure 31. Valve stem marked by coated O-rings.

Figure 32. Gland plate after testing.

Figure 33. O-ring shows signs of coating removal.

CHAPTER 5

CONCLUSION

The purpose of this research was to determine if the application of a metal coated O-ring using the PlasmaBond® process would reduce leakage from an upper O-ring seal of a 2 inch, pressure class 300, manual ball valve. A further purpose of this research was to determine if lubrication in conjunction with the metallic coating would reduce stem leakage.

Analysis of the research data resulted in failure to reject the null hypothesis in all but one instance. The one instance that the null hypothesis was rejected was for the 50 cycle PlasmaBond® test and the results only exceeded the objective criterion by a small margin. It was concluded, from the analysis, that this research does not support a claim that there is an improvement in leakage using the PlasmaBond® treatment alone or with lubrication.

The leak rate increased as testing progressed through the test sequences regardless of the treatment of the samples. It was concluded from this that the increase in leakage was caused by a variable that was dependent on the number of test cycles. Dynamic elastomeric seals have the unusual ability to wear harder metal surfaces (Martini 1984). While there were no visible signs of wear, it was conclude that a wear related mechanism was probably the cause of the increased leakage. The use of the random test sequence prevented a wear related variable from inducing an error into the analysis.

Finally, it could conclude from the plots of the interaction of the variables that the use of the PlasmaBond® treatment might improve leakage for unlubricated O-rings. The test of objective criteria failed to support this conclusion. While there may indeed be some reason to believe there was a reduction in leakage, the observed leakage increase associated with the progression through the test sequences prevented this conclusion. Despite the attempt to randomize the test sequences, all of the uncoated, unlubricated O-rings were tested at test sequence 11 or later.

CHAPTER 6

RECOMMENDATIONS

Concluded from this research that there was no immediate improvement in valve stem leakage at room temperature for a class 300, manual ball valve using the PlasmaBond® process on the elastomeric stem seals. Because this research did not include any aging or elevated temperature effects, this conclusion may not be true for elevated temperatures or prolonged periods where the carbon in the O-ring material could react with the metal surfaces of the gland and stem. Therefore, It is recommend that future research be conducted to test leakage from PlasmaBond® coated O-rings that have been installed for some period of time or at an elevated temperature.

APPENDIX A

HADAMARD MATRICES

0 Cycle Leakage

Trial	0	A 1	B 2	3	4	-AB 5	6	7	8	9	10	11	12	13	14	15
1	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
2	1.E-04	1.E-04	1.E-04	-1.E-04	-1.E-04	-1.E-04	1.E-04	-1.E-04	-1.E-04	1.E-04	1.E-04	-1.E-04	1.E-04	-1.E-04	1.E-04	1.E-04
3	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
4	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
5	6.E-05	-6.E-05	6.E-05	6.E-05	6.E-05	6.E-05	-6.E-05	-6.E-05	-6.E-05	6.E-05	-6.E-05	-6.E-05	6.E-05	6.E-05	-6.E-05	6.E-05
6	3.E-04	3.E-04	-3.E-04	3.E-04	3.E-04	3.E-04	3.E-04	-3.E-04	-3.E-04	-3.E-04	3.E-04	-3.E-04	-3.E-04	3.E-04	3.E-04	-3.E-04
7	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04
8	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
9	2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04
10	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
11	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04
12	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
13	1.E-04	-1.E-04	1.E-04	-1.E-04	-1.E-04	1.E-04	1.E-04	-1.E-04	1.E-04	-1.E-04	1.E-04	1.E-04	1.E-04	1.E-04	-1.E-04	-1.E-04
14	2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04
15	3.E-04	-3.E-04	-3.E-04	-3.E-04	3.E-04	-3.E-04	-3.E-04	3.E-04	3.E-04	-3.E-04	3.E-04	-3.E-04	3.E-04	3.E-04	3.E-04	3.E-04
16	2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04
Sum		-7.E-04	-5.E-04	-3.E-04	7.E-04	-5.E-04	7.E-04	-6.E-05	-3.E-04	-3.E-04	1.E-04	-5.E-04	-3.E-04	5.E-04	3.E-04	-1.E-04
Mean High																
-Mean Low		-8.E-05	-7.E-05			-7.E-05										
Variance				7.2E-09	2.7E-08		3.4E-08	2.3E-10	4.2E-09	7.2E-09	1.2E-09	1.3E-08	7.2E-09	1.3E-08	7.2E-09	1.2E-09

Ave Variance 1.0E-08

Ave Std Deviation 1.0E-04

Test Criterion	9.1E-05	Null Hypothesis	1	2	3
			Fail to Reject	**Fail to Reject**	**Fail to Reject**

10 Cycle Leakage

Trial		A	B			-AB										
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	2.E-05	2.E-05	-2.E-05	-2.E-05	-2.E-05	2.E-05	-2.E-05	-2.E-05	2.E-05	2.E-05	-2.E-05	2.E-05	-2.E-05	2.E-05	2.E-05	2.E-05
2	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04
3	2.E-05	2.E-05	2.E-05	2.E-05	-2.E-05	-2.E-05	-2.E-05	2.E-05	-2.E-05	-2.E-05	2.E-05	2.E-05	-2.E-05	2.E-05	-2.E-05	2.E-05
4	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
5	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
6	3.E-04	3.E-04	-3.E-04	3.E-04	3.E-04	3.E-04	3.E-04	-3.E-04	-3.E-04	-3.E-04	3.E-04	-3.E-04	-3.E-04	3.E-04	3.E-04	-3.E-04
7	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04
8	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
9	2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04
10	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
11	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04
12	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
13	9.E-05	-9.E-05	9.E-05	-9.E-05	-9.E-05	9.E-05	9.E-05	-9.E-05	9.E-05	-9.E-05	9.E-05	9.E-05	9.E-05	9.E-05	-9.E-05	-9.E-05
14	2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04
15	3.E-04	-3.E-04	-3.E-04	-3.E-04	3.E-04	-3.E-04	-3.E-04	3.E-04	3.E-04	-3.E-04	3.E-04	-3.E-04	3.E-04	3.E-04	3.E-04	3.E-04
16	1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04
Sum		-4.E-04	-4.E-04	-4.E-04	6.E-04	-6.E-04	1.E-03	1.E-05	-2.E-04	-2.E-04	4.E-04	-4.E-04	-3.E-04	4.E-04	6.E-04	5.E-05
Mean High																
-Mean Low		-4.E-05	-5.E-05			-8.E-05										
		Variance		9.51E-09	2.0E-08		5.64E-08	6.25E-12	2.76E-09	2.26E-09	9.51E-09	8.56E-09	3.91E-09	1.16E-08	2.33E-08	1.56E-10
		Ave Variance			1.23E-08											
		Ave Std Deviation			1.1E-04											

Test Criterion	9.95E-05	Null Hypothesis	1	2	3
			Fail to Reject	**Fail to Reject**	**Fail to Reject**

25 Cycle Leakage

Trial		A	B			-AB										
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	1.E-04	1.E-04	-1.E-04	-1.E-04	-1.E-04	1.E-04	-1.E-04	-1.E-04	1.E-04	1.E-04	-1.E-04	1.E-04	-1.E-04	1.E-04	1.E-04	1.E-04
2	3.E-04	3.E-04	3.E-04	-3.E-04	-3.E-04	-3.E-04	3.E-04	-3.E-04	-3.E-04	3.E-04	3.E-04	-3.E-04	3.E-04	-3.E-04	3.E-04	3.E-04
3	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
4	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
5	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
6	2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04
7	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04
8	3.E-05	3.E-05	-3.E-05	3.E-05	-3.E-05	3.E-05	3.E-05	3.E-05	3.E-05	-3.E-05	-3.E-05	-3.E-05	3.E-05	-3.E-05	-3.E-05	3.E-05
9	1.E-04	1.E-04	1.E-04	-1.E-04	1.E-04	-1.E-04	1.E-04	1.E-04	1.E-04	1.E-04	-1.E-04	-1.E-04	-1.E-04	1.E-04	-1.E-04	-1.E-04
10	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
11	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04
12	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
13	1.E-04	-1.E-04	1.E-04	-1.E-04	-1.E-04	1.E-04	1.E-04	-1.E-04	1.E-04	-1.E-04	1.E-04	1.E-04	1.E-04	1.E-04	-1.E-04	-1.E-04
14	2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04
15	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04
16	1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04
Sum		-3.E-04	-3.E-04	-5.E-04	7.E-05	-5.E-04	9.E-04	-3.E-04	-3.E-04	7.E-05	3.E-04	-1.E-04	-7.E-05	7.E-05	7.E-04	3.E-04
Mean High																
-Mean Low		-3.E-05	-4.E-05			-6.E-05										
			Variance	1.38E-08	3.06E-10		5.41E-08	4.56E-09	4.56E-09	3.06E-10	4.56E-09	1.06E-09	3.06E-10	3.06E-10	2.81E-08	6.81E-09
			Ave Variance		9.89E-09											
			Ave Std Deviation		9.9E-05											

Test Criterion	8.9E-05	Null Hypothesis	1	2	3
			Fail to Reject	**Fail to Reject**	**Fail to Reject**

50 Cycle Leakage

Trial		A	B			-AB										
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	9.E-05	9.E-05	-9.E-05	-9.E-05	-9.E-05	9.E-05	-9.E-05	-9.E-05	9.E-05	9.E-05	-9.E-05	9.E-05	-9.E-05	9.E-05	9.E-05	9.E-05
2	1.E-04	1.E-04	1.E-04	-1.E-04	-1.E-04	-1.E-04	1.E-04	-1.E-04	-1.E-04	1.E-04	1.E-04	-1.E-04	1.E-04	-1.E-04	1.E-04	1.E-04
3	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
4	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
5	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
6	2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04
7	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04
8	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
9	2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04
10	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
11	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04
12	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
13	2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04
14	2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04
15	3.E-04	-3.E-04	-3.E-04	-3.E-04	3.E-04	-3.E-04	-3.E-04	3.E-04	3.E-04	-3.E-04	3.E-04	-3.E-04	3.E-04	3.E-04	3.E-04	3.E-04
16	2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04	-2.E-04
Sum		-7.E-04	-5.E-04	-7.E-04	3.E-04	-5.E-04	7.E-04	-9.E-05	9.E-05	-3.E-04	1.E-04	-1.E-04	-3.E-04	5.E-04	3.E-04	-1.E-04
Mean High																
-Mean Low		-9.E-05	-6.E-05			-6.E-05										
			Variance	2.98E-08	6.01E-09		3.15E-08	5.06E-10	5.06E-10	6.01E-09	7.56E-10	7.56E-10	5.26E-09	1.5E-08	5.26E-09	7.56E-10
			Ave Variance		8.51E-09											
			Ave Std Deviation		9.2E-05											

Test Criterion	8.25E-05	Null Hypothesis	1	2	3
			Reject	**Fail to Reject**	**Fail to Reject**

100 Cycle Leakage

Trial	A	B			-AB											
	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	5.E-05	5.E-05	-5.E-05	-5.E-05	-5.E-05	5.E-05	-5.E-05	-5.E-05	5.E-05	5.E-05	-5.E-05	5.E-05	-5.E-05	5.E-05	5.E-05	5.E-05
2	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04
3	4.E-05	4.E-05	4.E-05	4.E-05	-4.E-05	-4.E-05	-4.E-05	4.E-05	-4.E-05	-4.E-05	4.E-05	4.E-05	-4.E-05	4.E-05	-4.E-05	4.E-05
4	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
5	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
6	3.E-04	3.E-04	-3.E-04	3.E-04	3.E-04	3.E-04	3.E-04	-3.E-04	-3.E-04	-3.E-04	3.E-04	-3.E-04	-3.E-04	3.E-04	3.E-04	-3.E-04
7	1.E-04	-1.E-04	1.E-04	-1.E-04	1.E-04	1.E-04	1.E-04	1.E-04	-1.E-04	-1.E-04	-1.E-04	1.E-04	-1.E-04	-1.E-04	1.E-04	1.E-04
8	1.E-05	1.E-05	-1.E-05	1.E-05	-1.E-05	1.E-05	1.E-05	1.E-05	1.E-05	-1.E-05	-1.E-05	-1.E-05	1.E-05	-1.E-05	-1.E-05	1.E-05
9	3.E-04	3.E-04	3.E-04	-3.E-04	3.E-04	-3.E-04	3.E-04	3.E-04	3.E-04	3.E-04	-3.E-04	-3.E-04	-3.E-04	3.E-04	-3.E-04	-3.E-04
10	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
11	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04	-2.E-04	2.E-04
12	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00	0.E+00
13	2.E-04	-2.E-04	2.E-04	-2.E-04	-2.E-04	2.E-04	2.E-04	-2.E-04	2.E-04	-2.E-04	2.E-04	2.E-04	2.E-04	2.E-04	-2.E-04	-2.E-04
14	1.E-04	-1.E-04	-1.E-04	1.E-04	-1.E-04	-1.E-04	1.E-04	1.E-04	-1.E-04	1.E-04	-1.E-04	1.E-04	1.E-04	1.E-04	1.E-04	-1.E-04
15	3.E-04	-3.E-04	-3.E-04	-3.E-04	3.E-04	-3.E-04	-3.E-04	3.E-04	3.E-04	-3.E-04	3.E-04	-3.E-04	3.E-04	3.E-04	3.E-04	3.E-04
16	1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04	-1.E-04
Sum		-1.E-04	-2.E-04	-6.E-04	5.E-04	-6.E-04	9.E-04	-2.E-04	2.E-04	-2.E-04	6.E-04	-5.E-04	-3.E-04	7.E-04	2.E-04	-1.E-04
Mean High																
-Mean Low		-1.3E-05	-2.8E-05			-7.3E-05										
			Variance	2.25E-08	1.56E-08		5.29E-08	2.5E-09	3.03E-09	2.5E-09	2.1E-08	1.69E-08	4.9E-09	2.89E-08	2.5E-09	6.25E-10
			Ave Variance		1.45E-08											
			Ave Std Deviation		1.2E-04											

Test Criterion	1.1E-04	Null Hypothesis	1	2	3
			Fail to Reject	**Fail to Reject**	**Fail to Reject**

APPENDIX B

TEST RESULTS

Test Sequence	Trial	Factors	Press (psig)	Temp (ºF)	Cycles	IS 3000IS (cc/sec)	GOW Mac 21-250 (divisions)	Stem Torque (ft-lbs.)
1	4	ab	504	73	0	0	0	N/A
1	4	ab	502	73	10	0	0	14
1	4	ab	498	74	25	0	0	16
1	4	ab	521	74	50	0	0	19
1	4	ab	514	75	100	0	0	23
2	8	a-	516	76	0	0	0	N/A
2	8	a-	512	76	10	0	0	20
2	8	a-	509	77	25	3.00E-05	15	22
2	8	a-	505	77	50	0	0	22
2	8	a-	501	77	100	1.00E-05	25	23
3	10	-b	502	70	0	0	0	N/A
3	10	-b	498	71	10	0	0	17
3	10	-b	518	72	25	0	0	19
3	10	-b	514	72	50	0	10	19
3	10	-b	510	72	100	0	0	22
4	3	ab	504	73	0	0	0	N/A
4	3	ab	522	72	10	2.00E-05	30	25
4	3	ab	522	73	25	0	10	26
4	3	ab	517	73	50	0	15	29
4	3	ab	515	74	100	4.00E-05	20	27
5	12	a-	500	73	0	0	0	N/A
5	12	a-	516	74	10	0	0	29
5	12	a-	514	73	25	0	0	25
5	12	a-	512	73	50	0	0	29
5	12	a-	508	74	100	0	0	29
6	5	-b	504	73	0	6.00E-05	45	N/A
6	5	-b	496	73	10	0	0	25

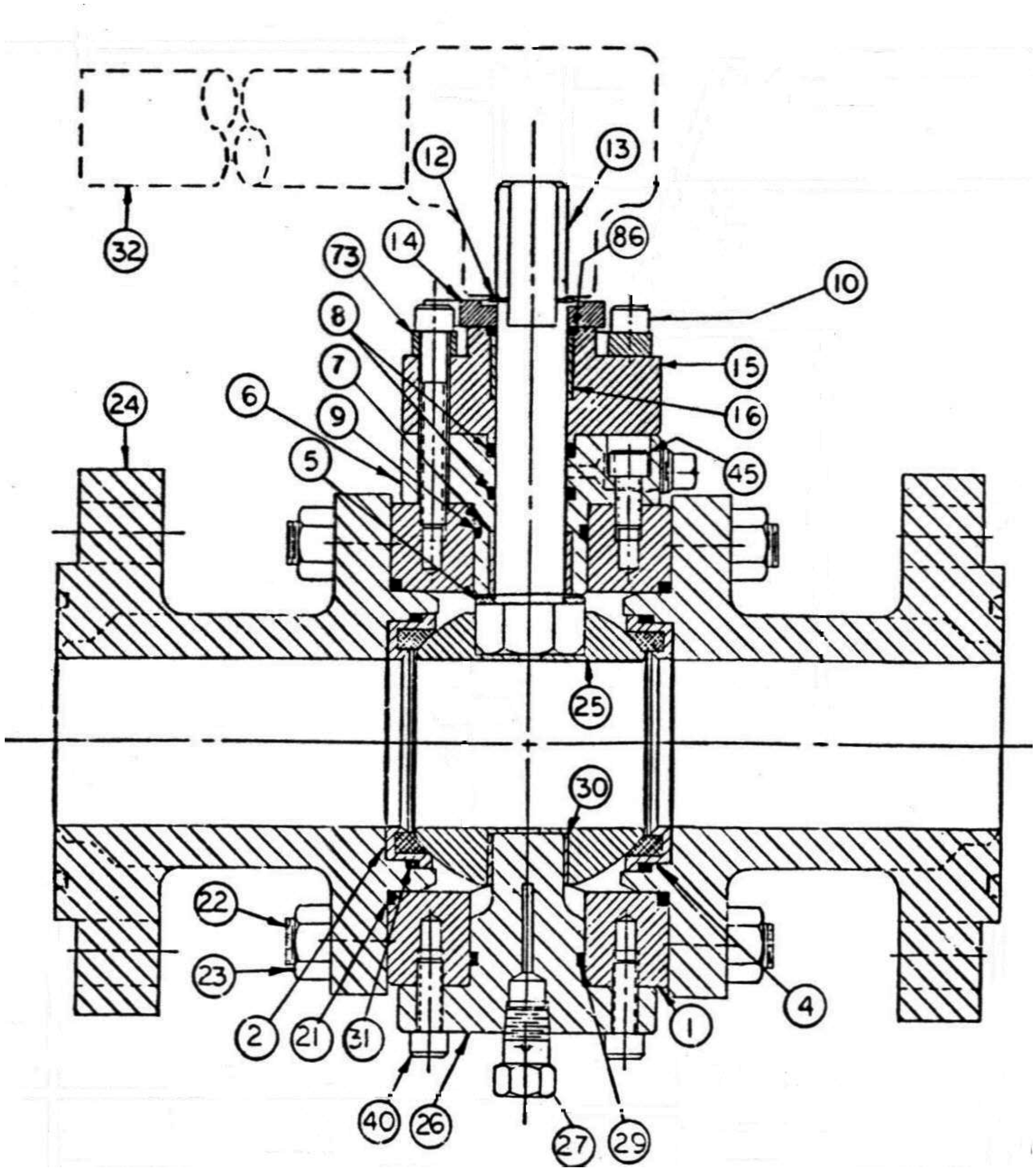
Test Sequence	Trial	Factors	Press (psig)	Temp (ºF)	Cycles	IS 3000IS (cc/sec)	GOW Mac 21-250 (divisions)	Stem Torque (ft-lbs.)
6	5	-b	496	72	25	0	0	27
6	5	-b	516	73	50	0	0	30
6	5	-b	502	73	100	0	30	27
7	1	a-	506	73	0	0	0	N/A
7	1	a-	502	73	10	2.00E-05	40	26
7	1	a-	507	74	25	1.00E-04	50	30
7	1	a-	508	70	50	9.00E-05	50	30
7	1	a-	506	75	100	5.00E-05	50	31
8	7	-b	509	74	0	2.00E-04	50	N/A
8	7	-b	500	75	10	2.00E-04	50	26
8	7	-b	496	74	25	2.00E-04	50	27
8	7	-b	525	75	50	2.00E-04	50	24
8	7	-b	514	76	100	1.00E-04	50	26
9	9	ab	510	76	0	2.00E-04	50	N/A
9	9	ab	516	75	10	2.00E-04	50	26
9	9	ab	514	76	25	1.00E-04	50	27
9	9	ab	512	76	50	2.00E-04	50	25
9	9	ab	512	77	100	3.00E-04	50	24
10	2	ab	509	76	0	1.00E-04	50	N/A
10	2	ab	504	76	10	2.00E-04	50	27
10	2	ab	500	78	25	3.00E-04	50	26
10	2	ab	498	78	50	1.00E-04	50	27
10	2	ab	496	79	100	2.00E-04	50	26
11	14	--	508	78	0	2.00E-04	50	N/A
11	14	--	507	77	10	2.00E-04	50	24
11	14	--	506	78	25	2.00E-04	50	25
11	14	--	505	78	50	2.00E-04	50	26

Test Sequence	Trial	Factors	Press (psig)	Temp (ºF)	Cycles	IS 3000IS (cc/sec)	GOW Mac 21-250 (divisions)	Stem Torque (ft-lbs.)
11	14	--	504	79	100	1.00E-04	50	28
12	11	--	500	72	0	2.00E-04	50	N/A
12	11	--	496	73	10	2.00E-04	50	26
12	11	--	512	74	25	2.00E-04	50	28
12	11	--	514	73	50	2.00E-04	50	27
12	11	--	512	74	100	2.00E-04	50	27
13	13	-b	508	74	0	1.00E-04	50	N/A
13	13	-b	500	74	10	9.00E-05	50	25
13	13	-b	516	74	25	1.00E-04	50	24
13	13	-b	508	75	50	2.00E-04	50	23
13	13	-b	502	75	100	2.00E-04	50	27
14	16	--	512	77	0	2.00E-04	50	N/A
14	16	--	512	77	10	1.00E-04	50	22
14	16	--	510	77	25	1.00E-04	50	23
14	16	--	508	78	50	2.00E-04	50	23
14	16	--	506	78	100	1.00E-04	50	22
15	6	a-	500	77	0	3.00E-04	50	N/A
15	6	a-	515	78	10	3.00E-04	50	23
15	6	a-	523	78	25	2.00E-04	50	26
15	6	a-	522	79	50	2.00E-04	50	22
15	6	a-	521	79	100	3.00E-04	50	23
16	15	--	508	79	0	3.00E-04	50	N/A
16	15	--	504	79	10	3.00E-04	50	21
16	15	--	503	79	25	2.00E-04	50	24
16	15	--	502	79	50	3.00E-04	50	22
16	15	--	502	80	100	3.00E-04	50	22

APPENDIX C

TEST VALVE

(Grove Valve 1994)



32
12
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ITEM	PART NAME	MATERIAL
1	BODY	STEEL
2	SPRING WASHER	SPRING STEEL
4	SEAT UNIT	STEEL & SYNTHETIC MAT’L
5	THRUST BEARING	STEEL & TEFLON
6	GLAND PLATE	STEEL
7	LOWER STEM BEARING	STEEL & TEFLON
8	STEM O-RING	GROVEX
9	GLAND PLATE O-RING	GROVEX
10	CAPSCREW (BEARING HOUSING)	ALLOY STEEL
12	RETAINING RING	STEEL
13	STEM	STEEL
14	STOP COLLAR	DUCTILE IRON
15	BEARING HOUSING	STEEL
16	STEM BEARING	STEEL & TEFLON
17	VENTED PLUG	STEEL
21	BODY O-RING	GROVEX
22	STUD	ALLOY STEEL
23	NUT	ALLOY STEEL
24	CLOSURE	CAST STEEL
25	BALL	STEEL
26	TRUNNION	STEEL
27	VENT PLUG ASSEMBLY	ALLOY STEEL
29	TRUNNION O-RING	GROVEX
30	TRUNNION BEARING	STEEL & TEFLON
31	SEAT O-RING	GROVEX
32	WRENCH	STEEL
40	CAPSCREW (TRUNNION)	ALLOY STEEL
45	CAPSCREW (GLAND PLATE)	ALLOY STEEL
22A	CAPSCREW (PLATE CLOSURE)	ALLOY STEEL
72	SPACER	STEEL
86	O-RING (WEATHER SEAL)	GROVEX

APPENDIX D

PLASMABOND® DATA SHEETS

TXU Electric PlasmaBond Center		PROCEDURE NO. PBP-0001
Administrative Control Procedure For The PlasmaBond Process	REVISION NO. 0	PAGE 9 OF 11

PlasmaBond Treatment Log
Pre-Treatment Inspection Section

Date: 1/10/2000 Application: Q / NQ (circle one)

PO/Document#: Lone Star Test Company Name: TXU (John Taylor)

Item ID#: (15) 1.065"OD x .1" O Rings Item ID#: Item ID#:
Item ID#: Item ID#: Item ID#:
Item ID#: Item ID#: Item ID#:
Item ID#: Item ID#: Item ID#:
Item ID#: Item ID#: Item ID#:
Item ID#: Item ID#: Item ID#:
Item ID#: Item ID#: Item ID#:
Item ID#: Item ID#: Item ID#:

Pre-Treatment Non-conforming Section

Item ID#: Item ID#: N/A Item ID#:
Comments: Resolution:

Coating Section

Metal Type Ti Lot#____ Total(gm) 1.1 Total # Filaments 2 Total Man-Hrs____
Metal Type Ni Lot#____ Total(gm) 1.4 Total # Filaments 2 Total Man-Hrs____
Metal Type Au Lot#____ Total(gm) 3.6 Total # Filaments 2 Total Man-Hrs____

Coating: SAT / UNSAT Initials / Date: W 1/10/2000
Comments: DC 50 RF 45

Post-Treatment Non-conforming Section

Item ID#: Item ID#: Item ID#:
Comments: N/A Resolution:
Notified (if UNSAT): Date:
Company: Phone: Date / Time:

PBP-0001-1

O-Ring Test for John Taylor .100D x 3.023L

O- Rings- 0.125 D x 3.30L
P.O.# Lone Star Test Customer Name TXU (John Taylor)

Designation	O-Ring Dia. (in)	O-Ring Length (in)	Collector Area (sq-cm)	Initial Weight (gm)	Final Weight (gm)	Weight Difference (gm)	Avg. Deposit (mg/sq-cm)	M&TE No. TU-7776 Cal Due Date
1	0.125	3.14159	7.9553007	1.4409	1.4459	0.005	0.629	_3_/_21/_01_
2	0.125	3.14159	7.9553007	1.427	1.4325	0.0055	0.691	

O- Rings- 0.100 D x 3.032L
P.O.# Lone Star Test Customer Name TXU (John Taylor)

Designation	O-Ring Dia. (in)	O-Ring Length (in)	Collector Area (sq-cm)	Initial Weight (gm)	Final Weight (gm)	Weight Difference (gm)	Avg. Deposit (mg/sq-cm)	M&TE No. TU-7776 Cal Due Date
1	0.1	3.032	6.1422329	0.7360	0.7382	0.0041136	0.670	_3_/_21/_01_
2	0.1	3.032	6.1422329	0.7356	0.7384	0.00471256	0.767	

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